Vol. II

TRANSCRIPT OF RECORD

Supreme Court of the United States

OCTOBER TERM, 1939

No. 9

STANDARD BRANDS, INCORPORATED,
PETITIONER,

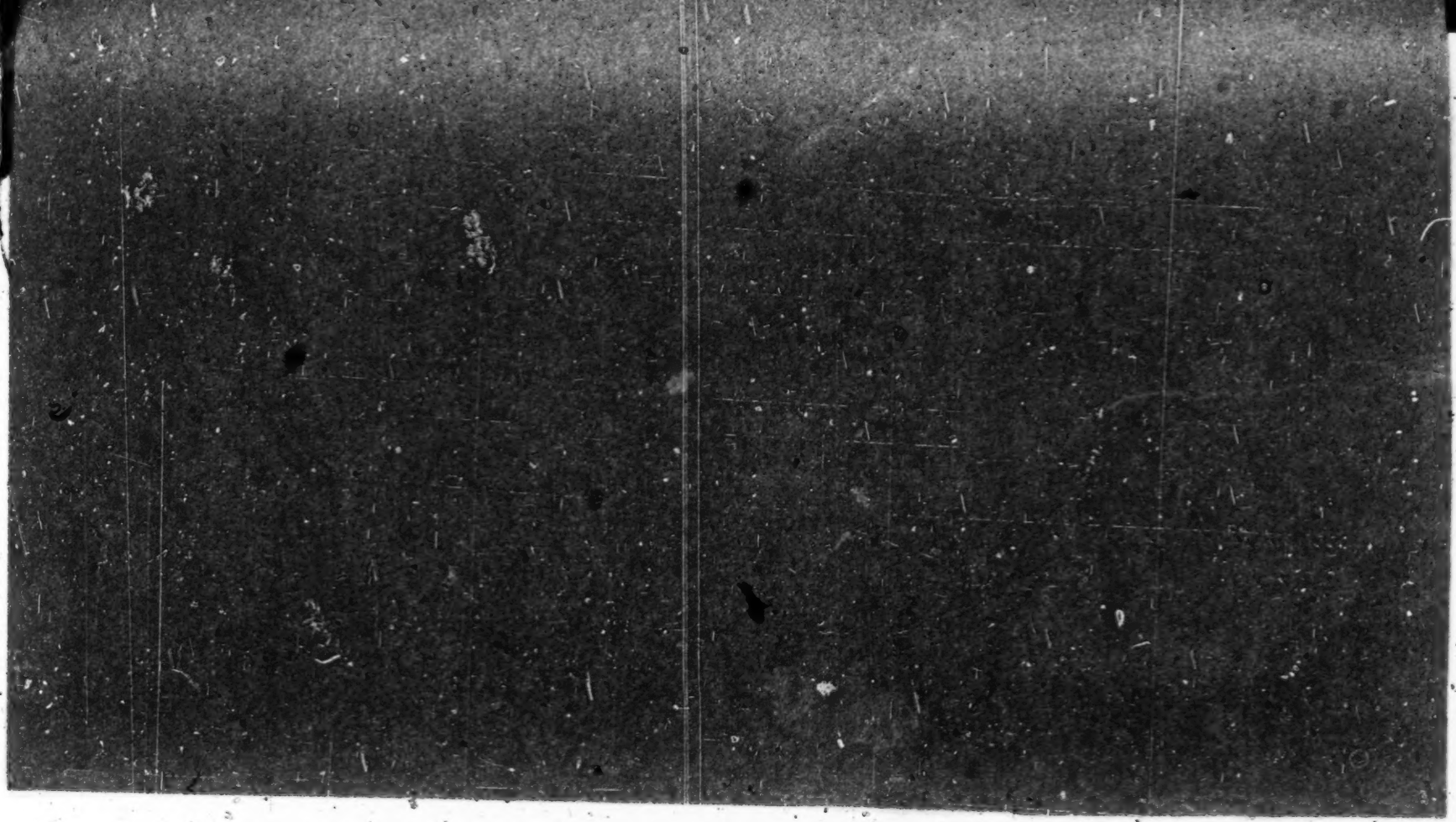
vs.

NATIONAL GRAIN YEAST CORPORATION

ON WRIT OF CERTIORARI TO THE UNITED STATES CIRCUIT COURT OF APEALS FOR THE THIRD CIRCUIT

PETITION FOR CERTIORARI FILED FEBRUARY 25, 1939.

CERTIORARI GRANTED MARCH 27, 1939.



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PLAINTIFF'S EXHIBITS

PLAINTIFF'S EXHIBIT P-1.

Patented Mar. 20, 1923. 1,449,103

UNITED STATES PATENT OFFICE.

FRIEDRICH HAYDUCK, OF WILMERSDORF, GERMANY, ASSIGNOR TO THE FLEISCHMANN COMPANY, OF NEW YORK, N. Y., A CORPORATION OF OHIO.

PROCESS FOR THE MANUFACTURE OF COMPRESSED YEAST.

No Drawing. Application filed October 30, 1920. Serial No. 420,830.

(GRANTED UNDER THE PROVISIONS OF THE ACT OF MARCH 3, 1921, 41 STAT. L., 1313.)

To all whom it may concern:

Be it known that I, FRIEDRICH HAYDUCK, a citizen of Germany, residing at Wilmersdorf, Germany, have invented certain new and useful Improvements in Processes for the Manufacture of Compressed Yeast and the Product (for which application for patent was filed as follows, Germany, March 15, 1915, Patent No. 303,251), of which the following is a specification.

This invention relates to a process for the manufacture of yeast, particularly bakers' yeast, by propagation in a solution containing sugar material and yeast nourishing inorganic salts.

An object of this invention is to provide a procedure for the manufacture of yeast on a large scale commercially, there being added to the nutrient solution which contains sugar material and yeast nourishing inorganic salts a neutralizing agent for neutralizing the deleterious excess of acidity.

Another object is to provide a good bakers' yeast in a convenient and economical manner.

In the production of yeast in accordance with the process described in German Patent No. 300,663, and in my corresponding co-pending application for United States Patent Serial No. 420,829, filed October 30, 1920, there takes place in solutions of sugar and inorganic salts, even when no bacterial infection sets in, a considerable increase in the acid content of the nutrient solution in which the yeast is produced. The resultant acidity is, for the most part, of an inorganic nature, and such inorganic acid is released from the inorganic salts used, the basic parts are used to a large extent as nutriment by the yeast, while the residual acid parts remain. This selective partial utilization by the yeast of components of inorganic salts present tends to result in an accumulation of acid products or free inorganic acids and tends increasingly to acidify the solution in which the yeast is being propagated or grown. Because of the presence of these free acids, the yeast, as can be seen by examination of the same with a microscope, is influenced unfavorably if the acids exert their action unrestrainedly upon the yeast for any considerable length of time.

I have now discovered that unexpectedly high yeast yields are obtained when conducting the nutrition of the yeast with the use of solutions containing sugar and yeast-nutrient inorganic salts in accordance with a process of the character exemplified in my aforementioned copending application Serial Number 420,829, if the acid components referred to as being set free during the yeast propagation and growth are neutralized either entirely or to such an extent that they no longer affect the yeast unfavorably. For such neutralization of the acid components, calcium carbonate may suitably be employed, but other basic or antacid substances such as antacid substances containing or supplying nitrogen can also be employed for the neutralization, for example ammonium carbonate.

A suitable yeast-nutrient solution for the propagation of yeast according to the method of my present invention may be formed as follows and as described in my aforesaid co-pending application Serial No. 420,829: 100 parts of sugar are dissolved in tap water together with 6 parts ammonium sulfate, 2 parts ammonium dihydrogenphosphate ($NH_4H_2PO_4$), 4 parts potassium sulfate, 2½ parts of gypsum and 2½ parts magnesium sulfate and the solution is made up to a total quantity of 6500 to 7000 parts with the requisite quantity of water, (all "parts" being stated by weight).

The process of my present invention may be executed in such manner, for example that calcium carbonate is added to the nutrient solution either at the beginning or fractionally during the process, to an amount corresponding to the sulfuric acid contained in and which may be set free from the ammonium sulfate used in the yeast growing process in case ammonium sulfate is employed as one of the principal inorganic yeast-nutrient salts. In the case of such employment of ammonium sulfate, in accordance with the molecular weight of the compounds involved, there may be suitably employed for every 10 parts by weight of ammonium sulfate (since the yeast tends to utilize for its nutrition the total basic part of this salt) up to 7½ parts by weight of calcium carbonate. Molasses may be used in my process as a suitable source of sugar for the yeast production and if molasses

naturally containing antacid substances is used as the sugar-containing raw material the quantity of calcium carbonate employed can be reduced materially, as the antacid substances present in such molasses neutralize a part of the free acid. Whether calcium carbonate or an antacid nitrogen-supplying substance or other neutralizing agent be used it will, of course, be employed in such amount as to neutralize whatever detrimental excess of free acid tends to develop in the nutrient medium during the yeast growth and as stated above it may be added fractionally in successive portions.

The technical advantages of my improved process hereinafter claimed can be seen from the following:

While in a process as described in my above-mentioned copending application for United States Patent No. 420,829, from 100 kilograms of sugar employed in the yeast-nutrient solution not more than about from 70 to 80 kilograms of compressed bakers' yeast is ordinarily obtained, there can be obtained when making use of the method including neutralization in accordance with the process of this present application, up to 160 kilograms of bakers' yeast from a nutrient solution containing 100 kilograms of sugar, and, if there is used a quick growing species of yeast, up to 300 kilograms of compressed yeast can be obtained from such a nutrient solution containing 100 kilograms of sugar, that is, therefore, from two to four times the yield obtained in the heretofore usual process employing cereal wort for the manufacture of yeast by the aeration method, calculated on the starch or available carbohydrate content of the raw materials, and up to approximately twice the yield, similarly calculated, obtained by my improved process as set forth in my copending application for United States patent Serial Number 420,829.

The neutralization of acids may also be advantageous in processes involving the fermentation of pure sugar solutions in the presence of inorganic salts in certain instances even when the obtainment of yeast is not intended.

It is known that there can be added to mashes in lactic fermentation, nitrogen-supplying antacid substances, as for instance ammonia, ammonium carbonate or ammonium carbamate, for the partial neutralization of the lactic acid progressively formed and for the augmentation of the yeast-assimilable nitrogen content of the mashes so that they may be utilized for subsequent yeast propagation.

It is also known that an addition of basic or antacid substances to some fermenting liquids stimulates the fermentation, the injurious action of the acids on the life of the yeast being inhibited. Furthermore, there has already been some theoretical discussion with respect to the possibility of obtaining yeast on an industrial scale from solutions containing sugar and inorganic salts. A practical embodiment of such a process, based upon my discoveries, is described in and broadly claimed in my copending application for United States patent Ser. No. 420,829 hereinabove referred to. An outstanding novel and technically important feature of my present invention as compared with the process described and claimed in my aforesaid copending application Serial No. 420,829 resides in the fact that by means of the careful neutralization of the aforesaid acid components set free are neutralized either entirely or to an extent that they no longer affect the yeast unfavorably whereby yeast yields may be obtained which have not been obtainable hitherto by any process and which could not have been predicted from the known facts.

While in the foregoing description I have given a detailed embodiment of my present invention, it will, of course, be apparent that the invention is not limited to the use of the particular substances employed as yeast nutrients but that my present invention is merely typified and illustrated by the example given and includes broadly all such processes as come within the scope of the claims hereinafter presented, bearing in mind the fact that substances such as fermentable sugars, added as such or in the form of molasses, and suitable yeast-nutrient salts as typified by those in the example given will be used, in accordance with my process and claims, in such aggregate quantities and in such proportions as are requisite for supplying in the proper proportions and in yeast-assimilable form or combination the necessary carbon, nitrogen, phosphorus and other essential yeast-constituent elements for the production of yeast in such quantities as are desired and preferably in the high yield hereinabove indicated as obtainable by the process of my present invention, when carried out with suitable aeration of the nutrient solution during the yeast production. It is known that the fermenting power and other desirable characteristics of yeast are to a certain degree dependent upon a sufficiency, in the nutrient solution in which the yeast is produced, of nitrogen-supplying substances and in the practice of my present invention such a quantity of ammonium salts and other substances will of course be ordinarily employed as will produce a compressed yeast having the well-known desirable nitrogen content and properties.

As is stated in my German Patent No. 300,663 and in my corresponding application for United States patent Serial No. 420,829, specially good results with respect

to the obtainment of high yields of yeast from nutrients of the types involved in the present application may be secured by employment of nutrient solutions which have a density of not more than 2° on the saccharometer.

Having described my invention, what I claim as new and desire to secure by Letters Patent is:—

1. The process of manufacturing yeast which comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nutrient solution containing essentially sugar material and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the nutrient solution during propagation, and during the period of propagation neutralizing the deleterious excess of such acidity.

2. The process of manufacturing yeast which comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nurtient solution containing essentially sugar material and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the nutrient solution during propagation, and during the period of propagation neutralizing the deleterious excess of such acidity by the addition of an innocuous antacid substance.

3. The process of manufacturing yeast which comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nutrient solution containing essentially sugar material and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the nutrient solution during propagation, and during the period of propagation neutralizing the deleterious excess of such acidity by the fractional addition of an innocuous antacid substance.

4. The process of manufacturing yeast comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nutrient solution containing essentially sugar material and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the nutrient solution during propagation, and during the period of propagation neutralizing the deleterious excess of such acidity by the addition of an innocuous antacid substance adapted to supply yeast assimilable nitrogen to the yeast.

5. The process of manufacturing yeast which comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nutrient solution containing essentially sugar material and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the nutrient solution during propagation, and during the period of propagation neutralizing the deleterious excess of such acidity by the addition of an innocuous carbonate.

6. The process of manufacturing yeast which comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nutrient solution containing essentially sugar material and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the nutrient solution during propagation, and during the period of propagation neutralizing the deleterious excess of such acidity by the addition of ammonium carbonate.

7. The process of manufacturing yeast which comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nutrient solution containing essentially sugar material and yeast nourishing inorganic salts including a compound containing yeast assimilable nitrogen which is adapted to be at least partially assimilated by the yeast and from which during such assimilation components are liberated which tend increasingly to acidify the nutrient solution during propagation, and during the period of propagation neutralizing the deleterious excess of such acidity.

8. The process of manufacturing yeast which comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nutrient solution containing essentially sugar material and yeast nourishing inorganic salts including an inorganic ammoniacal compound adapted to supply at least a material portion of yeast assimilable nitrogen required by the yeast during propagation, said salts being of a character such that components are liberated which tend increasingly to acidify the nutrient solution during propagation, and during the period of propagation neutralizing the deleterious excess of such acidity.

9. The process of manufacturing yeast which comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nutrient solution containing essentially sugar material and yeast nourishing inorganic salts including ammonium sulphate which is adapted to be at least partially assimilated by the yeast and from which during such assimilation components are liberated which tend increasingly to acidify the nutrient solution during propagation, and during the period of propagation neutralizing the deleterious excess of acidity.

10. The process of manufacturing bakers' yeast which comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nutrient solution being of a dilution corresponding to not

more than 2° on the saccharometer and containing essentially sugar material and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the nutrient solution during propagation, and during the period of propagation neutralizing the deleterious excess of such acidity.

11. The process of manufacturing bakers' yeast which comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nutrient solution being of a dilution corresponding to not more than 2° on the saccharometer and containing essentially sugar material and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the nutrient solution during propagation, and during the period of propagation neutralizing the deleterious excess of such acidity by the fractional addition of an innocuous antacid compound.

12. The process of manufacturing yeast which comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nutrient solution containing essentially molasses and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the nutrient solution during propagation, and during the period of propagation neutralizing the deleterious excess of such acidity.

13. The process of manufacturing yeast which comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nutrient solution containing essentially molasses and yeast nourishing inorganic salts including a compound containing yeast assimilable nitrogen which is adapted to be at least partially assimilated by the yeast and from which during such assimilation components are liberated which tend increasingly to acidify the nutrient solution, and during the period of propagation neutralizing the deleterious excess of such acidity.

14. The process of manufacturing bakers' yeast which comprises preparing a yeast nutrient solution and propagating yeast therein with aeration, said yeast nutrient solution consisting essentially of molasses and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the yeast nutrient solution during propagation, and during the period of propagation neutralizing the deleterious excess of acidity by the addition of an innocuous antacid compound.

15. As an article of manufacture, a yeast product adapted for baking purposes, of which substantially all of the principal constituents have been assimilated during propagation in a nutrient solution consisting of essentially sugar material and yeast nourishing inorganic salts from which components are released tending increasingly to acidify the nutrient solution during propagation, the deleterious excess of such acidity being neutralized during the period of propagation by the addition of an innocuous antacid substance.

In testimony whereof, I have affixed my signature.

FRIEDRICH HAYDUCK.

Certificate of Correction.

It is hereby certified that in Letters Patent No. 1,449,103, granted March 20, 1923, upon the application of Friedrich Hayduck, of Wilmersdorf, Germany, for an improvement in "Processes for the Manufacture of Compressed Yeast," errors appear in the printed specification requiring correction as follows: Page 1, line 44, after the word "acids" insert the word *in;* page 3, line 24, claim 2, for the word "nurtient" read *nutrient;* and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 19th day of June, A. D., 1923.

[SEAL.]

WM. A. KINNAN,
Acting Commissioner of Patents.

Patented Mar. 20, 1923. 1,449,105

UNITED STATES PATENT OFFICE.

FRIEDRICH HAYDUCK, OF WILMERSDORF, GERMANY, ASSIGNOR TO THE FLEISCHMANN COMPANY, OF NEW YORK, N. Y., A CORPORATION OF OHIO.

LOW-ALCOHOL YEAST PROCESS.

No Drawing. Application filed October 30, 1920. Serial No. 420,832.

(GRANTED UNDER THE PROVISIONS OF THE ACT OF MARCH 3, 1921, 41 STAT. L., 1313.)

To all whom it may concern:

Be it known that I, FRIEDRICH HAYDUCK, a citizen of Germany, residing at Wilmersdorf, Germany, have invented certain new and useful Improvements in Low-Alcohol Yeast Processes (for which an application for patent was filed in Germany March 16, 1915, Patent No. 300,662), of which the following is a specification.

This invention relates to a process for the manufacture of yeast and particularly to a process for manufacturing a compressed bakers' yeast in which very little alcohol is produced as a by-product, and has for its object an improved procedure for manufacturing yeast in a more convenient and economical manner than heretofore.

In the manufacture of yeast according to the usual aeration process, large quantities of alcohol are generated, the amount of which is to some extent dependent on the sugar available for the yeast.

Efforts have been made to produce economical conditions under which not only an increase of the yeast yield from the raw material used would be insured, but a simultaneous diminution of the alcohol yield would be obtained. It has therefore already been suggested to dilute the wort to such an extent that the alcohol produced from the sugar could supposedly again be assimilated by the yeast as nourishment. The process disclosed in the German patent to Rainier, 10135, endeavors to reach this goal by adding the nutrient required for the yeast (sugar and peptone), in a manner which would produce this result, i. e., by adding the peptones continuously and the sugar at intervals, as required, and whenever analysis showed the need thereof, stating that in keeping the concentration of the sugar very low the formation of alcohol is prevented and the sugar is used only as a nutrient. In the operations set forth by this patent, the very low concentration of the sugar solution which is added from time to time to the fermenting wort, requires the use of large tanks and apparatus to handle the large volume of liquid. However, the increase in operating costs connected with this high dilution is partly compensated for by the advantage of the high yield in the yeast.

In the process described hereinafter and forming the subject of this invention, the goal of obtaining a high yeast yield with little or no formation of alcohol is reached by the use of worts of normal quality and concentration, the wort being added to the yeast continuously and at a rate such that no large excess of alcohol will be formed, and the yeast can assimilate most of the alcohol which is so formed.

The process may be, for example, executed in the following manner:

A wort adapted for yeast propagation (having, for example, a gravity of 12° Balling) is prepared according to the procedure commonly followed in the compressed yeast industry. A part of this wort is placed in the fermenting vat and is highly diluted (for example, to about 1° Balling), yeast is added thereto and it is aerated. Thereupon the remainder of the concentrated wort is added slowly and substantially continuously throughout a protracted period of time and at a rate such that not only the alcohol which may have been formed from the quantity of sugar present in the diluted portion of the wort, but also any alcohol which may be formed from the sugar which is present in the added wort, can be assimilated immediately by the yeast. If arrangements are made to carry on the aeration process for ten hours, the addition of the wort may be effected for about seven to eight hours under continuous aeration and the aeration may be subsequently continued for two to three hours without the addition of any wort. The quantity of the diluted wort which is originally available for the yeast is so chosen, for example, that the mean content of the entire wort employed would be 3° to 5° Balling.

As compared with the obtaining of yeast from worts of very small concentration, the process described above offers the advantage that the yeast is constantly given new supplies of nutrient constituents necessary for its propagation by the continuous addition of concentrated wort, and that thereby as a result of the continual consumption of the nutrients by the yeast, there is always a wort concentration, by means of which the nutrients and the result-

ant substances are absorbed most efficaciously and quickly by the yeast, that is to say, there is offered in this manner of procedure through the peculiar distribution of the wort, the advantage of great dilution, while the concentration of the wort (calculated for the entire quantity) remains normal.

In the process in accordance with German Patent 10135, the intermittent or continuous addition of the peptones or the intermittent successive addition of sugar to the liquid containing the yeast is provided for by making use of the high dilution, which favors the growth of the yeast at the expense of the formation of alcohol. But, aside from the fact that this process requires constant supervision of the conditions of nutrition obtaining in the liquid, as sugar and peptone can only be added separately and as required, this process does not provide for work with normally concentrated liquids, nor does it disclose the propagation of yeast in a wort of usual constitution, the resultant wort used for the propagation of yeast having a peptone sugar ratio of 2/1. Moreover, the operation of this process requires the estimation of the sugar and peptone content prior to further additions of the respective solutions containing these nutrients, and it therefore lacks the simple operation characterizing the process herein described, which does not differ in any way, in regard to the production of the wort and the amount of labor required, from the usual procedure followed in the manufacture of aerated yeast.

In the manufacture of compressed yeast, the starting of the primary, undiluted wort with yeast, and the adding of the secondary wort, which is being constantly diluted, is a known method, the concentrated wort from the mash process being filtered from the draff and led into the fermenting vat. After a portion of this wort has been run into the vat, the seed yeast is added, and while the thus seeded portion is being aerated the remainder of the concentrated mash is filtered and run into the fermenting vat together with a stream of water, the sparge water being later run into the fermenting vat in the same manner. The amount of water which is constantly run into the vat as the wort is added is so regulated that after all of the concentrated mash wort, the sparge water and the added water have been put into the vat so as to fill the same, the wort will have the required concentration. The conditions for propagating the yeast in the best possible manner are considered to be fulfilled, when all of the filtered wort and sparge water are added in the shortest possible time. It will thus be seen that the propagation begins in a relatively concentrated solution, and that the concentration thereof is decreased gradually, partly because the nutrient substances are being consumed by the yeast and partly because the addition of water and sparge water gradually reduce the initial concentration of the wort. In this procedure, in which, therefore, the opposite principle is followed, such large quantities of alcohol are generated from the very start, that no appreciable assimilation of the alcohol can take place.

Having described my invention what I claim as new and desire to secure by Letters Patent is—

1. A process of propagating yeast which comprises, initiating propagation of yeast in a dilute nutrient solution containing all essential yeast nutrients, aerating the solution, and substantially continuously adding during the period of propagation, a solution containing all essential yeast nutrients.

2. A process of propagating yeast which comprises, preparing a wort containing all essential yeast nutrients in solution, initiating propagation of yeast in a diluted portion of said wort, and substantially continuously adding during the period of propagation, the wort of higher concentration.

3. A process of propagating yeast which comprises, preparing a wort containing all essential yeast nutrients in solution, initiating propagation of yeast in a diluted portion of said wort, aerating the diluted portion, and substantially continuously adding during the period of propagation, the wort of higher concentration.

4. A process of propagating yeast with a relatively low yield of alcohol which comprises preparing a wort containing all essential yeast nutrients in solution, initiating propagation of yeast in a diluted portion of said wort, aerating the diluted portion, and substantially continuously adding during the period of propagation, a wort of higher concentration, at a rate such that the yeast may propagate and substantially all of the alcohol which may be formed is assimilated by the yeast.

5. A process of propagating yeast with a relatively low yield of alcohol which comprises, preparing a wort containing all essential yeast nutrients in solution, initiating propagation of yeast in a diluted portion of said wort, aerating the diluted portion, and slowly and substantially continuously adding during the period of propagation, the wort of higher concentration.

6. A process of propagating yeast with a relatively low yield of alcohol which comprises, preparing a wort containing all essential yeast nutrients in solution, initiating propagation of yeast in a diluted portion of said wort, aerating the diluted portion and slowly and substantially continuously adding the wort of high concentration, which

addition is effected until near the end of the period of propagation.

7. A process of propagating yeast which comprises, preparing a dilute wort and a relatively concentrated wort, each containing all essential yeast nutrients in solution, initiating propagation of yeast in the dilute wort, aerating the dilute wort and substantially continuously adding during the period of propagation, the wort of higher concentration.

8. A process of propagating yeast with a relatively low yield of alcohol which comprises, preparing a wort containing all essential yeast nutrients in solution, initiating propagation of yeast in a diluted portion of said wort, aerating the diluted portion, and substantially continuously adding during the period of propagation, the wort of higher concentration at a rate such that the yeast may propagate and substantially all of the alcohol which may be formed is assimilated by the yeast.

9. A process of propagating yeast with a relatively low yield of alcohol which comprises, preparing a wort containing all essential yeast nutrients in solution, initiating propagation of yeast in a diluted portion of said wort, aerating the diluted portion, and substantially continuously adding during the period of propagation, the wort of higher concentration at a rate such that the concentration of the dilute wort does not rise above that at which substantially all of the alcohol which may be formed, will be assimilated by the yeast.

10. A process of propagating yeast with a relatively low yield of alcohol which comprises, preparing a wort containing all essential yeast nutrients in solution, initiating propagation of yeast in a highly diluted portion of said wort, aerating the diluted portion, and substantially continuously adding during the period of propagation, the wort of higher concentration at a rate such that the concentration of the dilute wort remains substantially constant, whereby substantially all of the alcohol which may be formed is assimilated.

In testimony whereof, I have affixed my signature.

FRIEDRICH HAYDUCK.

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PLAINTIFF'S EXHIBIT P-3.

Patented Mar. 20, 1923. **1,449,106**

UNITED STATES PATENT OFFICE.

FRIEDRICH HAYDUCK, OF WILMERSDORF, GERMANY, ASSIGNOR TO THE FLEISCHMAN COMPANY, OF NEW YORK, N. Y., A CORPORATION OF OHIO.

LOW-ALCOHOL YEAST PROCESS.

No Drawing. Application filed October 30, 1920. Serial No. 420,833.

(GRANTED UNDER THE PROVISIONS OF THE ACT OF MARCH 3, 1921, 41 STAT. L., 1313.)

To all whom it may concern:

Be it known that I, FRIEDRICH HAYDUCK, citizen of Germany, residing at Wilmersdorf, Germany, have invented certain new and useful Improvements in Low-Alcohol Yeast Processes (for which application for patent was filed in Germany March 19, 1915, Patent No. 303,222), of which the following is a specification.

This invention relates to a process for the manufacture of yeast and particularly to a process for manufacturing a compressed bakers' yeast in which very little alcohol is produced as a by-product and has for its object an improved procedure for manufacturing yeast in a more convenient and economical manner than heretofore.

In accordance with the process protected by my German Patent 300,662 and set forth in my corresponding copending application for United States Patent Serial No. 420,832, filed Oct. 30, 1920, a high yeast yield is obtained at the expense of the production of alcohol, in worts of normal concentration, by starting the growth of the yeast in a very weak solution and thereafter adding concentrated wort slowly and substantially continuously, whereby, as a result of the constant consumption of the nutrients and the resultant alcohol on the part of the yeast, in spite of the addition of concentrated wort, there is always a sufficiently low concentration of nutrients and yeast conversion products. For the carrying out of this process the use of yeast wort of normal quality is contemplated.

In the process described hereinafter, which constitutes the subject-matter of my present invention, this peculiar distribution of the nutrient liquid is applied to worts, which are prepared principally from sugar or sugar containing substances (for example, molasses), and inorganic salts. In the production of yeast from such saccharifierous solutions, containing mineral salts as yeast nutrients, the difficulty has hitherto always been encountered that the yeast cannot stand such high salt concentrations as are required for its full nourishment in yeast nutrient solutions of heretofore usual sugar content, and that it was therefore necessary, to work either with greatly diluted nutrient liquids or solutions or with solutions containing a disproportionately small quantity of inorganic yeast-nutrient salts. The latter procedure, however, results in correspondingly small yeast yields because of the deficiency of most essential substances or elements for which the yeast was dependent upon the inorganic salts.

By applying the hereinafter described process of "adding," the results are attained that owing to the appropriate gradual addition of nutrients at the desired rate, there is not only avoided an accumulation of alcohol, but also deleterious accumulation of nutrient salts is avoided, as the salts, as well as substantially all resulting alcohol which may be formed, are immediately assimilated by the yeast. The manipulative operation of the process is the same as described in German Patent 300,662 and in the aforesaid corresponding copending application for United States Patent Serial No. 420,832.

For the preparation of the nutrient solutions there are used, for instance, sugar and yeast-nutrient inorganic salts (ammonium, potassium and magnesium combined with phosphoric acid, sulphuric acid, etc.). This process permits of the use of the salts in a proportion up to half as much total salts as sugar. When using ammonium sulphate as a nitrogen source, the rapid assimilation of the nutrient salts supplied for the yeast can be recognized by the surprisingly great increase in acid content (sulphuric acid ions set free) in the nourishing liquid. The deleterious excess of this liberated acid can be eliminated by the addition of alkalies or salts having an alkaline reaction in accordance, for example, with the process of my copending application for United States Patent Serial No. 420,830.

The process, for example, may be suitably carried out in the following manner:

To 100 parts of sugar there are used: 40 parts of ammonium sulphate, 9 parts acid phosphate of potassium, 1 part magnesium sulphate, to which is to be added, about 30 parts calcium carbonate for the neutralization.

To 100 parts molasses (with 50 per cent

sugar content), there are used: 10 parts ammonium sulphate, 10 parts superphosphate (having 18% water soluble phosphoric acid), 0.5 parts magnesium sulphate, to which is to be added about 4 parts calcium carbonate.

The raw nutrient materials are dissolved in sufficient water so that the sugar content of the solution amounts to about 10 percent. As soon as the yeast is brought into the fermenting vat with a part of the nutrient solution, which has been previously diluted as will be hereinafter described aeration and the addition of the remaining nutrient solution is started. Carrying out the process of the present invention in a manner similar to that described in my copending application for United States Patent Serial No. 420,832, the seed yeast may be added in the first instance to a solution of a strength of about 1° Balling and containing also a proportionate amount of nutrient salts which are used, and thereafter during the growth of the yeast the solution containing about 10% of sugar and a proportionate quantity of the nutrient salts, may be added slowly and substantially continuously so as to maintain the concentration of about the solution in which the yeast is propagating approximately constant or at a slightly greater or less concentration during the main portion of the period of propagation. The calcium carbonate or chalk is either all added at the beginning or, in accordance with the increase in acidity, gradually, to the fermenting vat. One can also distribute the yeast in the beginning in water in the fermenting vat, and then gradually and substantially continuously add the nutrient solution as described. It will be seen that the liquid in which the yeast is suspended should be non-toxic to the yeast. After all the nutrient solution has been added, the aeration is continued and the propagation is concluded in the well known manner, the yeast being thereafter separated from the finally attenuated nutrient solution wherein it has been grown.

Although my process is not limited to any particular duration of the period of yeast propagation, which may vary within considerable limits and may comprise such periods as are deemed proper or found most desirable in accord with the facilities and results desired in a particular yeast manufacturing establishment, it has been found, as stated in my German Patent No. 300,662, that if an aeration period totalling about 10 hours is employed, the gradual addition of the nutrient solution to the aerated solution as described may cover a period of 7 to 8 hours followed by a period of 2 to 3 hours aeration after this gradual addition is completed. The rate of addition and the duration of the process may be determined in accordance with tests so that the concentration remains approximately constant and at such a degree of dilution that accumulation of alcohol in the solution is substantially prevented, owing to the fact that in such dilute solution any alcohol which may be formed is again assimilated by the yeast, and an especially efficient utilization of the nutrient substances for obtaining a high yield of yeast in the process is thus attained.

Having described my invention what I claim as new and desire to secure by Letters Patent, is:

1. A process of propagating yeast which comprises preparing a nutrient solution comprising essentially sugar material and yeast nourishing inorganic salts, suspending seed yeast in a non-toxic aqueous liquid, aerating the liquid, and substantially continuously adding the nutrient solution.

2. A process of propagating yeast with a relatively low yield of alcohol which comprises preparing a nutrient solution, exclusive of cereal material, comprising essentially sugar material and yeast nourishing inorganic salts, suspending seed yeast in a non-toxic aqueous liquid, aerating the liquid, and substantially continuously adding the nutrient solution.

3. A process of preparing yeast with a relatively low yield of alcohol which comprises suspending seed yeast in a non-toxic aqueous liquid, aerating the liquid, and substantially continuously adding thereto sugar material and yeast nourishing inorganic salts at a rate such that the concentration of the aerated liquid does not arise above that at which substantially all of the alcohol which may be formed is assimilated by the yeast.

4. A process of propagating yeast with a relatively low yield of alcohol which comprises suspending seed yeast in a non-toxic aqueous liquid, aerating said liquid, substantially continuously adding thereto sugar material and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the solution during propagation, which addition is at a rate such that the concentration of the aerated liquid does not arise above that at which substantially all of the alcohol which may be formed is assimilated by the yeast, and during the period of propagation neutralizing the deleterious excess of such acidity.

5. A process of propagating yeast with a relatively low yield of alcohol which comprises suspending seed yeast in a non-toxic aqueous liquid, aerating said liquid, substantially continuously adding thereto sugar material and yeast nourishing inorgan[illegible]

salts from which components are liberated which tend increasingly to acidify the solution during propagation, which addition is at a rate such that the concentration of the aerated liquid does not rise above that at which substantially all of the alcohol which may be formed is assimilated by the yeast, and during the period of propagation neutralizing the deleterious excess of such acidity, by the fractional addition of an innocuous antacid substance.

6. A process of propagating yeast with a relatively low yield of alcohol which comprises preparing a nutrient solution comprising essentially sugar material and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the solution during propagation, suspending seed yeast in a non-toxic aqueous liquid, aerating the liquid, substantially continuously adding the nutrient solution to the aerated liquid, and adding during the period of propagation an innocuous antacid substance adapted to neutralize at least a substantial portion of such acidity.

7. A continuous process of propagating yeast with a relatively low yield of alcohol which comprises preparing a nutrient solution, exclusive of cereal material, comprising essentially sugar material and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the solution during propagation, suspending seed yeast in a non-toxic aqueous liquid, aerating the liquid, thereafter substantially continuously adding the nutrient solution to the aerated liquid, and during the period of propagation neutralizing the deleterious excess of such acidity.

8. A process of propagating yeast with a relatively low yield of alcohol which comprises preparing a nutrient solution comprising essentially sugar material and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the solution during propagation, suspending seed yeast in a non-toxic aqueous liquid, aerating the liquid, substantially continuously adding the nutrient solution to the aerated liquid, and during the period of propagation neutralizing at least a substantial portion of such acidity, by the fractional addition of an innocuous antacid substance.

9. A process of propagating yeast with a relatively low yield of alcohol which comprises initiating propagation of yeast in a highly dilute nutrient solution consisting essentially of water, sugar material, and yeast nourishing inorganic salts, aerating the solution, and gradually and substantially continuously adding thereto a more concentrated aqueous solution of sugar material and yeast nourishing inorganic salts, the total amount of yeast nourishing inorganic salts used in the process amounting to about one-half of the total amount of sugar used.

10. A process of propagating yeast with a relatively low yield of alcohol which comprises preparing a nutrient solution containing essentially sugar material and yeast nourishing inorganic salts, the total amount of yeast nourishing inorganic salts used being about one-half of the total amount of sugar used, highly diluting a portion of the nutrient solution, initiating propagation of yeast therein with aeration, and during the period of propagation slowly and substantially continuously adding the concentrated solution.

11. A process of propagating yeast with a relatively low yield of alcohol which comprises preparing a nutrient solution containing essentially sugar material and yeast nourishing inorganic salts from which components are liberated which tend increasingly to acidify the nutrient solution during propagation, the total amount of yeast nourishing inorganic salts used being about one-half of the total amount of sugar used, highly diluting a portion of the nutrient solution, initiating propagation of yeast therein with aeration, during the period of propagation slowly and substantially continuously adding the concentrated solution, and neutralizing a substantial portion of such acidity by the addition of an innocuous antacid substance.

12. A process of propagating yeast with a relatively low yield of alcohol which comprises initiating propagation of yeast in a dilute nutrient solution comprising essentially sugar material and yeast nourishing inorganic salts, aerating the solution, and adding thereto substantially continuously during the period of propagation a solution containing sugar material and yeast nourishing inorganic salts at a rate such that the concentration of the solution in which the yeast is propagating is maintained substantially constant.

13. A process of propagating yeast with a relatively low yield of alcohol which comprises initiating propagation of yeast in a dilute nutrient solution comprising essentially sugar material and yeast nourishing inorganic salts, from which components are liberated which tend increasingly to acidify the nutrient solution during propagation, aerating the solution, during the period of propagation adding thereto substantially continuously sugar material and yeast nourishing inorganic salts, at a rate such that the concentration of the solution in which the yeast is propagating is maintained substantially constant, and during the period of propagation neutralizing a substantial portion of such acidity by the addition of an innocuous antacid substance.

14. The process of propagating yeast which comprises initiating the yeast growth in a dilute aqueous solution comprising essentially sugar material and yeast nourishing inorganic salts, aerating the solution and adding thereto gradually and continuously, and at least during the major portion of the period of yeast propagation therein, a yeast nutrient solution containing essentially sugar material and yeast nourishing inorganic salts, the salt content of the solutions being approximately one-half the sugar content.

15. A process of propagating yeast which comprises initiating the propagation of yeast in a highly dilute nutrient solution comprising essentially water, molasses and yeast nourishing inorganic salts from which, during propagation, components are liberated which tend increasingly to acidify the nutrient solution, aerating the solution, and during the period of propagation adding to the solution a more concentrated aqueous solution of molasses and yeast nourishing inorganic salts, and sufficient quantities of an innocuous antacid substance to neutralize at least a substantial portion of such acidity.

16. A process of propagating yeast which comprises initiating the propagation of yeast in a dilute aqueous yeast nutrient solution containing sugar material and yeast nourishing inorganic salts from which, during propagation, components are liberated which tend increasingly to acidify the nutrient solution, aerating the solution, adding to the nutrient solution containing the yeast, during at least a portion of the period of propagation, additional sugar and material and yeast nourishing inorganic salts, the proportion of total inorganic salts employed during the entire period of propagation being approximately one-half the total quantity of sugar employed, and during the period of propagation, neutralizing at least a portion of such acidity.

In testimony whereof, I have affixed my signature.

FRIEDRICH HAYDUCK.

Certificate of Correction.

It is hereby certified that in Letters Patent No. 1,449,106, granted March 20, 1923, upon the application of Friedrich Hayduck, of Wilmersdorf, Germany, for an improvement in "Low-Alcohol Yeast Processes," errors appear in the printed specification requiring correction as follows: Page 2, line 12, after the word "solution" strike out the comma and insert the same to follow the word "described" in line 13; same page, line 29, strike out the word "about"; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 29th day of May, A. D., 1923.

[SEAL.]

KARL FENNING,
Acting Commissioner of Patents.

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PLAINTIFF'S EXHIBIT P-4.

Patented Mar. 20, 1923. 1,449,109

UNITED STATES PATENT OFFICE.

FRIEDRICH HAYDUCK, OF WILMERSDORF, GERMANY, ASSIGNOR TO THE FLEISCHMANN COMPANY, OF NEW YORK, N. Y., A CORPORATION OF OHIO.

LOW-ALCOHOL YEAST PROCESS.

No Drawing. Application filed October 30, 1920. Serial No. 420,836.

(GRANTED UNDER THE PROVISIONS OF THE ACT OF MARCH 3, 1921, 41 STAT. L., 1313.)

To all whom it may concern:

Be it known that I, FRIEDRICH HAYDUCK, a citizen of Germany, residing at Wilmersdorf, Germany, have invented certain new and useful Improvements in Low-Alcohol Yeast Processes (for which application for patent was filed in Germany April 23, 1915, Patent No. 304,243), of which the following is a specification.

This invention relates to a process for the manufacture of yeast, and particularly to a process for manufacturing a compressed "bakers" yeast in which very little alcohol is produced as a by-product and has for its object an improved procedure for manufacturing yeast in a more convenient and economical manner than heretofore.

In accordance with the process described in my German Patent 303,222, and in my corresponding application for United States Patent, Serial No. 420,833, filed October 30, 1920, a particularly high yeast yield is obtained, at the expense of the production of alcohol by a process in which nutrient solutions, which have been produced from sugar or sugar-containing substances and yeast-nutrient inorganic salts are added so gradually during the yeast propagation or growth that the yeast can immediately assimilate the nutrients which may be present together with substantially all of the alcohol which may be formed, under constant aeration.

In the preferred embodiment of my invention described and claimed in my aforesaid application, Serial No. 420,833, the seed yeast is added either to water or to a very dilute nutrient solution and there is gradually and substantially continuously added a relatively concentrated solution of yeast-nutrient materials, as for example, 10% of sugar together with a proportionate amount of suitable inorganic yeast nutrient salts which may be as high as up to one-half of the amount of sugar. Such solutions may have the following proportions:

Sugar 100 parts, ammonium sulphate 40 parts, acid potassium phosphate 9 parts, magnesium sulphate 1 part, calcium carbonate, for neutralization about 30 parts and sufficient water to form a solution containing about 10% of sugar: or

Molasses (containing about 50% sugar) 100 parts, ammonium sulphate 10 parts, Superphosphate (having 18% water soluble phosphoric acid) 10 parts, magnesium sulphate 0.5 parts, calcium carbonate about 4 parts and sufficient water to form a solution containing about 10% sugar.

In the above process I have further discovered that it is necessary for obtainment of otherwise unobtainable high yields of yeast to neutralize or bind the acid components set free from the inorganic salts by the yeast by the addition of alkalies or alkaline salts. The acid components, which are here set free and must be neutralized, represent in regard to the manufacture of yeast, as I have discovered, a largely harmful ballast.

As I have further discovered, a distinct industrial advantage is obtained if it is possible to use the alkalies themselves for the growth of yeast. In this case not only is there a conservation, at least in part, of the acids which are commercially required for the production of such nutrient salts as are used in the process of my aforesaid copending application Serial No. 420,833, but also there is an economizing of the alkalies which are commercially used to neutralize the said acids in the manufacture of the salts, and which in my process are required to neutralize the continuously liberated acid components in the manufacture of yeast. As is also apparent from my German Patent No. 304,242, and my corresponding application for United States patent, Serial No. 420,837, filed October 30, 1920, such a process can be carried out with suitable free alkalies in an assuredly successful manner.

I have now found that such a process in regard to the use of alkalies or salts having an alkaline reaction can be carried out most effectively, if a small quantity of such inorganic nutrient salts are added at the very beginning to that portion of the nutrient solution to which is added the pitch or seed yeast, and from which the yeast in the process of utilization of certain portions of the salts sets free acid ions. In this case the added alkalies neutralize and are in turn neutralized by the acid so produced, thereby making the nutrient solution safe for the yeast.

The process for example is carried out in the following manner: The nutrients required for the yeast (sugar and inorganic

salts, but exclusive of ammonium salts or other source of inorganic nitrogen) are first dissolved in water to form a solution containing approximately 10% of sugar material. A part of this solution, after being properly diluted with water, in general accordance with the process of my aforesaid copending application Serial No. 420,833 (modified as particularly described in the present application i. e., by the omission of the ammonium salts and substance having an alkaline reaction) is mixed with some ammonium sulphate and set on with seed yeast. The remainder of the nitrogenous substance required for supplying necessary yeast assimilable nitrogen for the yeast, is added in the form of free ammonia (ammonia water), to the relatively concentrated nutrient solution, which is gradually and substantially continuously added to the more dilute nutrient solution during propagation of the yeast. The addition is regulated preferably so that the ammonia can always be neutralized to full extent by the acids set free by the yeast. With such regulation a condition of not more than a limited acidity which is adapted for the yeast growth is maintained in the solution wherein the yeast is grown.

The process of adding ammonium salts to mashes or worts for the nourishment of the yeast, and of neutralizing the lactic acid formed by lactic fermentation in mashes prior to the fermentation by the yeast, by means of alkalies, especially ammonia, is known. See, for example the United States patent to Alfred Pollak, No. 1,123,920, issued January 5, 1915. In contrast to this, the process of my present application has for its object and comprises in its specific embodiment particularly the addition of alkalies or salts having an alkaline reaction to the nutrient as yeast nourishment, for the production of a high yield of yeast of good quality and in this process one and the same quantity of inorganic acids (repeatedly neutralized and reliberated from their salts during the yeast propagation or growth) constantly neutralizes the added free alkalies or salts having an alkaline reaction as they are gradually added with the concentrated wort. The alkaline substance thus gradually added is, as above described, preferably a substance such as ammonia or ammonia water which not only serves to neutralize acidity developed from the selective partial utilization of the inorganic yeast nutrient salts present but also serves to supply yeast-assimilable nitrogen requisite for the production of large yields of yeast of good quality.

The quantity of ammonium sulfate which, as above described, is suitably employed in preparing the nutrient solution may be such a quantity as is proportionate to the other inorganic salts which are at the time of its introduction present in the relatively dilute solution in which the yeast growth is taking place or is to take place. That is to say, if, for example, the yeast growth is started in a solution containing 1% of sugar (as described in my above-mentioned copending application Serial No. 420,833 on which the invention of my present application is in part based), a proportionate quantity of ammonium sulfate may suitably be used approximating one-fifth to four-tenths this amount of sugar (in accord with the exemplary formulas hereinbefore given) in which case the yeast-nutrient solution would contain initially 0.2% to 0.4% ammonium sulfate together with the proportionate quantities of non-nitrogenous yeast-nutrient substances. Thereafter, during the course of yeast propagation, the yeast-nutrient solution is aerated and the nutrients consumed by the yeast are replenished (so gradually as to permit utilization of any alcohol as formed and to prevent any substantial accumulation of alcohol in the nutrient solution) by the addition of a more concentrated solution which may as hereinbefore described contain for example about 10% sugar and proportionate amounts of the other yeast-nutrient salts. The procedure of my present invention, which discloses the progressive addition of an antacid substance supplying yeast assimilable nitrogen (e. g., ammonia water) to a dilute wort containing a salt or salts from which, during the period of propagation, components are liberated which tend to increasingly acidify the solution in which the yeast is propagated, differs from that of my copending application Serial No. 420,833, which shows the progressive addition of an acid-liberating nitrogen-supplying salt or salts, for example, ammonium sulphate, the deleterious excess of acid being neutralized by non-nitrogenous neutralizing agent. The use of an antacid nitrogenous substance disclosed in the present process serves to prevent deleterious increase of acidity during the yeast production and, as I have discovered, enables high yields of good quality yeast to be obtained, while at the same time effecting a saving in the amount of nutrient salts used.

Since the ammonia water or similar substance having an alkaline reaction and supplying yeast-assimilable nitrogen serves in the present process as the acid-neutralizing agent, it is of course apparent as is pointed out above that the calcium carbonate used in the typical illustrative formulas hereinbefore given and taken from my copending application for United States patent Serial No. 420,833 will not be used as described in

my said copending application but that the ammonia water or equivalent substance herein referred to will be used in the manner and for the purposes described.

Having described my invention, what I claim as new and desire to secure by Letters Patent is:—

1. A continuous process of propagating yeast which comprises, initiating propagation of yeast in a dilute nutrient solution containing a compound which is at least partially assimilable by yeast and from which, while being so assimilated, components are liberated which tend increasingly to acidify the nutrient solution, aerating the solution, and adding thereto during the process of propagation yeast nutrient materials, including a sufficient amount of a substance having an alkaline reaction and containing yeast assimilable nitrogen to neutralize substantially all of such acidity.

2. A continuous process of propagating yeast which comprises, initiating propagation of yeast in a dilute nutrient solution containing a compound which is at least partially assimilable by yeast and from which, while being so assimilated, components are liberated which tend increasingly to acidify the nutrient solution, aerating the solution, and adding thereto during the process of propagation yeast nutrient materials, including a substance having an alkaline reaction and containing yeast assimilable nitrogen, at a rate such as to maintain the concentration of the solution substantially constant and to neutralize substantially all of such acidity.

3. A continuous process of propagating yeast with a relatively low yield of alcohol which comprises, initiating propagation of yeast in a highly dilute nutrient solution containing a compound which is at least partially assimilable by yeast and from which, while being so assimilated, components are liberated which tend increasingly to acidify the nutrient solution, aerating the solution, and adding thereto during the process of propagation a more concentrated solution of yeast nutrient materials, including a substance having an alkaline reaction and containing yeast assimilable nitrogen, at a rate such as to maintain a concentration of the solution substantially constant and to neutralize substantially all of such acidity.

4. A continuous process of propagating yeast which comprises, initiating propagation of yeast in a dilute nutrient solution containing a compound which is at least partially assimilable by yeast and from which, while being so assimilated, components are liberated which tend increasingly to acidify the nutrient solution, aerating the solution, and adding thereto during the process of propagation a more concentrated solution of yeast nutrient materials, including a sufficient amount of ammonium hydrate to neutralize substantially all of such acidity.

5. A continuous process of propagating yeast with a relatively low yield of alcohol which comprises, initiating propagation of yeast in a highly dilute nutrient solution containing a compound which is at least partially assimilable by yeast and from which, while being so assimilated, components are liberated which tend increasingly to acidify the nutrient solution, aerating the solution, and adding thereto during the process of propagation a more concentrated solution of yeast nutrient materials including ammonium hydrate, at a rate such as to maintain the concentration of the solution substantially constant and to neutralize substantially all of such acidity.

6. A continuous process of propagating yeast with a relatively low yield of alcohol which comprises, preparing a dilute nutrient solution containing sugar material and an ammonium compound from which yeast may assimilate nitrogen and from which, during such assimilation, components are liberated which tend increasingly to acidify the nutrient solution, initiating propagation of yeast therein with aeration, and substantially continuously adding during the period of propagation a nutrient solution containing sugar material and a compound having an alkaline reaction and containing yeast assimilable nitrogen, at a rate such that the concentration of the aerated solution is maintained approximately constant and substantially all of such acidity is neutralized.

7. A continuous process of propagating yeast with a relatively low yield of alcohol which comprises, preparing a dilute nutrient solution containing sugar material and an ammonium compound from which yeast may assimilate nitrogen and from which, during such assimilation, components are liberated which tend increasingly to acidify the nutrient solution, initiating propagation of yeast therein with aeration, and substantially continuously adding during the period of propagation a nutrient solution containing sugar material and ammonium hydrate, at a rate such that the concentration of the aerated solution is maintained approximately constant and substantially all of such acidity is neutralized.

8. A continuous process of propagating yeast with a relatively low yield of alcohol which comprises, preparing a dilute nutrient solution containing sugar material and an ammonium compound from which yeast may assimilate nitrogen and from which, during such assimilation, components are liberated which tend increasingly to acidify the nutrient solution, initiating propagation of yeast therein with aeration.

and substantially continuously adding during the period of propagation a more concentrated nutrient solution containing sugar material and a compound having an alkaline reaction and containing yeast assimilable nitrogen, the addition being made at such a rate, and the relative proportions of the ammonium compound originally present in the dilute solution, the sugar material and the compound having an alkaline reaction, being such, that substantially all of such acidity is neutralized and the concentration of the solution in which the yeast is propagating is maintained approximately constant, whereby practically all alcohol which may be formed is assimilated by the yeast.

In testimony whereof, I have affixed my signature.

FRIEDRICH HAYDUCK.

PLAINTIFF'S EXHIBIT P-5.

Patented June 12, 1928. 1,673,735

UNITED STATES PATENT OFFICE.

ROBERT L. CORBY, OF NEW YORK, AND WILHELM H. F. BÜNRIG, OF MOUNT VERNON, NEW YORK, ASSIGNORS TO THE FLEISCHMANN COMPANY, OF NEW YORK, N. Y., A CORPORATION OF OHIO.

PROCESS FOR THE MANUFACTURE OF YEAST.

No Drawing. Application filed August 16, 1926. Serial No. 728,867

This invention relates to a process for the manufacture of yeast, especially baker's yeast, and more particularly to an improved method of carrying out a process of manufacturing yeast in which at least a portion of the yeast nutrient is added after the propagation of yeast has begun.

An object of this invention is to provide an improved procedure for the manufacture of yeast in an efficient, convenient and economical manner.

In its more specific aspects one of the objects of this invention is to provide a method of carrying out a process of manufacturing yeast in which the amounts of the added constituents are regulated in such a manner that the capacity of fermenters and other propagating apparatus is materially increased and large yields of yeast are obtained.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

The invention accordingly comprises the several steps and the relation and order of one or more of such steps with respect to each of the others thereof, which will be exemplified in the process hereinafter disclosed, and the scope of the application of which will be indicated in the claims.

In the art of yeast manufacture there has been a constant endeavor to obtain a yeast having better baking qualities and at the same time to increase the quantity of yeast which may be obtained from a given amount of materials and thereby decrease the net production cost.

Nutrient solutions for the propagation of yeast may be of a type prepared from all cereal material, from sugar material, together with suitable yeast nourishing inorganic compounds such, for example, as compounds containing yeast-assimilable nitrogen and phosphorus adapted to be utilized during yeast growth, or from a combination of the materials of these two types of nutrient solutions. The amount of yeast to be obtained by propagation in any of the above types of nutrient solutions is somewhat dependent upon the environment prevailing in the propagating vat or fermenter, which environment involves various factors such as the amount of aeration, the temperature and the density of the nutrient solution.

In the prior practice of yeast propagation, particularly in nutrient solutions containing sugar material and yeast-nutrient inorganic compounds, that is, a solution of the "sugar-salt" type, the control of the environment has been carried to a comparatively high order and fluctuations in the acidity of the solution were carefully minimized, suitable corrective agents being added whenever the desired limits were exceeded. For example, ammonium hydroxide has been used as an agent to depress undue acidity, particularly released acidity, while a suitable acid of an organic or inorganic nature, such as for example phosphoric, sulphuric or lactic acid has been used when necessary to maintain the acidity.

In order adequately to control the temperature it has been customary to provide the fermenter with a suitable cooling means, for example, an interiorly disposed cooling coil. During a portion of the propagation there may be a tendency for the temperature to rise in the fermenter. In consequence a gradually increasing circulation of the cooling medium in the cooling coil was generally practiced.

The condition of the nutrient solution has thus been controlled by making suitable readings at regular intervals and by employing when necessary suitable corrective measures, as the conditions indicate.

A nutrient solution of relatively high dilution, especially in the use of the so-called sugar-salt type of solutions, has been found to be preferable, such dilution having been attained either by initial dilution of the entire nutrient solution or by suspending the seed yeast in a diluted portion thereof or other aqueous liquid non-toxic to yeast and slowly and substantially continuously adding the remaining and more concentrated solution. Such last mentioned type of process is known alternately as a "continuous", an "addition", or a "continuous addition" process. In either case, since the entire amount of the dilute nutrient solution is present in

the fermenter at the end of the process, relatively large fermenters have been required in order to obtain a given yield of yeast.

In the practice of the present invention, an environment is created and substantially maintained which is favorable to the propagation of large yields of yeast, at the same time using smaller apparatus than was heretofore possible with the use of the very dilute nutrient solutions now generally deemed preferable. This is achieved by the use of a fermenter, in which propagation is started, containing initially only a portion of the nutrient material from which a given batch of yeast is to be manufactured. During the period of propagation further nutrient material is added in a manner conducive to the prolific multiplication of yeast in the fermenter, as more fully set forth hereinafter.

To this end the nutrient solution is first prepared in a relatively concentrated condition. In case all the nutrient material is not contained initially in the prepared solution, supplemental nutrient material may be added, some to the portion first placed in the fermenter and the balance during the supplying of the solution run in during the course of propagation. The nutrients to be added thus may if desired be in solution.

In the preparation of the nutrient solution it should be the object to bring it to a condition favorable to yeast propagation. In the case of solutions of the sugar-salt type, this involves particularly the treatment of the sugar material in a manner so as to remove as far as practicable, all foreign material which may have a deleterious influence upon the yeast. For example, where molasses is the sugar material to be employed, this preliminary treatment is preferably such as to produce a clear, rapidly filtering solution giving a clear filtrate. Such treatment may comprise any suitable method and may involve heating, filtering and the addition of one or more clarifying agents.

A portion of the nutrient solution when prepared is placed in the fermenter and a properly tempered diluent, for example, water, both hot and cold, run in until the dilution, temperature and density are brought initially to the points adapted for the prolific multiplication of yeast after which the fermenter is preferably stocked with a relatively large amount of suitable seed yeast. The balance of the prepared solution which is in a relatively concentrated condition is placed in a supply reservoir so that it can be run into the fermenter during the period of propagation in such quantities and at such times as desired, the solution in the fermenter being continuously aerated during substantially the whole of such period.

Prolific propagation of yeast is here induced throughout the propagating period by regulating the addition of nutrients to the fermenter in such a manner as highly to stimulate the propagation of yeast without unduly raising the concentrations of the nutrient materials in solution at any time to a point which tends to become toxic to the yeast. The rate here practiced, however, is substantially in excess of that heretofore possible because of the stimulated activity of yeast. A materially increased activity of the yeast is attained at the beginning by the employment of temperature conditions within the fermenter which have heretofore been deemed impracticable. In the heretofore known processes of manufacturing yeast, the fermenter has been stocked while the fermenter is being filled and somewhat before the final dilution has been attained, and at a temperature of from 22°–25° C., and during the first few hours of the propagation the temperature has been allowed gradually to rise to a maximum of 30° C. In the present process, however, the temperature initially employed during a portion of at least one of the stages of propagation is much higher than heretofore and may be for example from 30°–35° C. or even up to the thermal death point of the yeast in a sugar solution. Moreover, contrary to the usual practice, this temperature is gradually lowered during the first hours of the stage of propagation until a temperature of 30° C. has been reached and is thereafter maintained approximately constant, the interval of time during which the high temperature is maintained being inversely proportional to the temperature.

As the propagation progresses, the presence in the nutrient solution both of the yeast cells themselves and the residual substances in the nutrient solution, either of an unassimilated or eliminated character, tend to increase the density and the toxicity generally prevailing in the nutrient solutions, whereas the relative concentrations of the nutrients present may or may not have changed. As propagation is here instituted under conditions particularly favorable to large yields of yeast, certain of the starting conditions are, in accordance with the practice of this invention, periodically approximately restored at intervals whenever such undesirable conditions are reached. For example, fluctuation in the acidity may be compensated from hour to hour by the addition of suitable antacid reagents, particularly those containing yeast assimilable nitrogen, and at intervals generally of several hours, "addition" is diminished or stopped, a substantial portion of the content of the fermenter is withdrawn and thereafter water, both warm and cold, is again added to the fermenter until certain of the starting conditions, particularly dilution, are substantially restored.

As the fermenter is not completely

drained, there remains, of course, a very substantial amount of yeast in the fermenter which serves as seed yeast for the next succeeding stage of the propagation. Furthermore, if desired, in order to increase the amount of seed yeast present, a portion of the yeast separated or pressed from the preceding stage may be added at the beginning of any subsequent stage. The total period of propagation practiced in this way may continue for from 12 to 24 or more hours. The interval at which yeast is to be drawn off, will, of course, preferably be some submultiple of the total period of propagation, such as for example, from 4 to 6 or 8 hours as conditions warrant.

It will be perceived that by this intermittent drawing off, fermenters of less total capacity may be employed than was heretofore possible, for a given yield of yeast. The reservoir, however, is preferably proportioned to contain the nutrient which is to be supplied during each stage of the propagating period. It will also be perceived that since the reserve reservoir contains the nutrient for the regulated additions in a relatively concentrated form, that less space is occupied by the main mass than would be required if it were diluted in toto in a fermenter. This process therefore includes the regulated supplying of further nutrient from an outside source to a fermenter and at suitable intervals drawing off yeast preferably accompanied by temporary diminution or cessation of "addition". Yeast is separated from the withdrawn solution cooled and ultimately pressed in the usual manner. Large yields of yeast, for example, yields approximating 85% and over, based on the sugar material employed, (assuming such sugar material to contain 50% fermentable sugars) may be obtained in the practice of this process. By the utilization of the principles set forth a greater amount of yeast may be produced from a given amount of material in a given time than was heretofore practical. The following is an example of the manner in which the process of this invention may be practiced:

The desired quantity of sugar material such as molasses of any suitable type for example, cane or beet molasses or a mixture thereof is treated in any suitable manner so as to yield a clear, rapidly filtering solution. A suitable treatment is for example one in which the molasses is clarified by the use of commercial calcium acid phosphate, ("super-phosphate") with or without the addition of a small amount of ammonia, the mixture being heated moderately. For example, a batch of 10,000 pounds of molasses may be diluted slightly, and, if desired, compounds of ammonia added thereto, the temperature raised to 60°–70° C., and about 1000 pounds of super-phosphate added, whereupon the mixture may be filtered through a suitable filter preferably with the accompanying use of "filter-cel" or the like. The resulting solution will have an acidity of about 5° or over (calculated as the number of cc. of normal soda necessary to neutralize 100 cc. thereof), and a concentration of about 15° to 20° Balling, such concentrations of the prepared nutrient solution will hereafter be referred to by the term "relatively concentrated".

A suitable portion of this prepared solution, for example up to about 15%, is run directly into the fermenter while the balance is pumped to a supply reservoir. Enough suitably tempered water is added to the fermenter to obtain a density of approximately 1°–2° Balling and a temperature of approximately 33° C., the acidity being adjusted if necessary to a suitable concentration such as, for example, 0.6°. A few minutes prior to the time at which the final dilution is reached, the seed yeast is added, preferably to an amount of from 20–27%, based on the sugar material (on the basis of 50% fermentable sugars) to be used during the first stage of propagation. The nutrient solution having been prepared and stocked, propagation is instituted with fairly high aeration, the temperature being slowly lowered until at about the end of the fourth hour it has reached about 30° C. and is thereafter maintained approximately constant, at least throughout any given stage of propagation.

During the period of propagation, the reserve nutrient solution is lowly and substantially continuously added in amounts such that the density is maintained substantially constant, such amounts being larger than those heretofore deemed practical and varying from hour to hour from approximately 2% up to 10%. Toward the end of and between each stage of the total period of propagation such "addition" is either largely diminished or entirely suspended in order to obtain a full utilization of the nutrient materials. For example, in a total fermentation time of 14 hours, no addition is made during the seventh hour or during the last hour and the rate of addition is considerably diminished during the sixth hour. Moreover, between the stages of the propagation period, for example, during the seventh hour while the "addition" has ceased, aeration is diminished and the yeast containing solution is withdrawn from the fermenter until for example about 10–70% of the quantity present at the end of the preceding hour remains, the withdrawal being regulated in accordance with the salt concentration of the liquid in the fermenter. The yeast containing solution thus withdrawn may be immediately separated and is desired a portion of the sepa-

rated yeast may be returned to the fermenter to act as seed yeast for the next succeeding stage or if desired the withdrawn solution may be used in a subsequent stage of propagation in a manner similar to the use of the residue in the fermenter. After such withdrawal sufficient water, both cold and hot, is added to the fermenter to maintain the temperature substantially constant, and again to bring the content of the fermenter up to approximately the initial volume of the diluted portion, whereupon "addition" and higher aeration are resumed.

When necessary either at the beginning of any stage of propagation or from hour to hour during the process, accompanying the additions from the storage reservoir, there is added to the fermenter supplemental nutrient materials adapted to regulate the acidity of the solution and to supply for example, yeast-assimilable nitrogen, preferably in the form of ammonium compounds. This may be accomplished by the selective addition of suitable ammonium compounds of an organic or inorganic nature, such as for example, aqua ammonia, ammonium carbonate, ammonium sulphate, ammonium phosphate, ammonium lactate and the like, it being desirable to maintain the acidity throughout the process approximately constant and to have present at the beginning of each stage of propagation a nitrogen concentration of approximately 0.06%, the concentration of the latter substance being allowed to fall gradually during each stage of the propagation in order to insure an efficient utilization thereof. For example, in the above described process there are added during each stage of the propagation a total of approximately 160 liters of aqua ammonia and 90 pounds of ammonium sulphate. Also regulated amounts of a suitable acid may be added if, and when necessary. In one aspect, therefore, the process may be considered as comprising the preparation of a yeast nutrient solution, the initiating of propagation of yeast with aeration in a diluted portion thereof, slowly and substantially continuously adding thereto yeast-nutrient materials to supply the deficiencies of the solution and including material to control the acidity, periodically concurrently diminishing or interrupting the addition of nutrients, lowering the aeration and withdrawing a portion of the yeast containing liquid, and thereafter diluting the residue thereof with suitably tempered water and then continuing the propagation with resumption of higher aeration and addition of nutrients.

By the use of the process, a total yield of yeast of approximately 85% and over (calculated on the basis of raw materials containing 50% fermentable sugars) may be obtained, such yeast having very desirable properties in that it presses well, is of a light color, and has good keeping qualities and excellent baking strength.

It will be observed that in carrying out the above process, a more efficient utilization of the apparatus is attained, in that at least double quantities of materials can be passed through a fermenter in a given time with a consequent saving in the amount of air and other ancillary materials and apparatus used for the propagation of yeast, while at the same time increasing the yield and quality of the yeast produced.

Since certain changes may be made in carrying out the above method without departing from the scope of the invention, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

It is also to be understood that the following claims are intended to cover all of the generic and specific features of the invention herein described, and all statements of the scope of the invention which, as a matter of language, might be said to fall therebetween.

Having described our invention, what we claim as new and desire to secure by Letters Patent, is:

1. A process of manufacturing yeast by propagation with aeration, which comprises suspending seed yeast in a non-toxic liquid, aerating the liquid, adding yeast nutrients to the liquid in regulated amounts during the propagating period, periodically withdrawing a portion of the yeast containing liquid, diluting the residue thereof and then continuing the propagation therein with addition of nutrients.

2. A process of manufacturing yeast by propagation with aeration, which comprises suspending seed yeast in a non-toxic aqueous liquid, aerating the liquid, adding yeast nutrients to the liquid in regulated amounts during the propagating period, periodically concurrently diminishing the addition of nutrients, lowering the aeration, and withdrawing a portion of the yeast containing liquid and thereafter diluting the residue thereof and continuing the propagation therein with resumption of higher aeration and addition of nutrients.

3. A process of manufacturing yeast by propagation with aeration, which comprises suspending seed yeast in a non-toxic aqueous liquid, aerating the liquid, adding yeast nutrients to the liquid in regulated amounts during the propagating period, periodically withdrawing a portion of the yeast containing liquid, diluting the residue thereof and continuing the propagation therein with addition of nutrients, the temperature of the liquid in which the seed yeast is sus-

pended and the temperature maintained during the first few hours only of a period of propagation being above 30° C.

4. A process of manufacturing yeast by propagation with aeration, which comprises suspending seed yeast in a non-toxic aqueous liquid, aerating the liquid, adding yeast nutrients to the liquid in regulated amounts during the propagating period, periodically concurrently diminishing the addition of nutrients, lowering the aeration, and withdrawing a portion of the yeast containing liquid, and thereafter diluting the residue thereof and continuing the propagation therein with resumption of higher aeration and addition of nutrients, the temperature of the liquid in which the seed yeast is suspended and the temperature maintained during a portion of the period of propagation being above 30° C.

5. A process of manufacturing yeast, which comprises starting propagation with a suspension of seed yeast in a fermenter partially filled with a non-toxic aqueous liquid, aerating the liquid, adding yeast nutrients to the liquid in regulated amounts, during the propagating period, periodically withdrawing a portion of the yeast containing liquid, diluting the residue thereof to approximately the volume of the liquid initially in the fermenter and then continuing the propagation therein with the accompanying addition of yeast nutrients.

6. A process of manufacturing yeast, which comprises starting propagation with a suspension of seed yeast in a fermenter partially filled with a dilute nutrient solution, aerating the solution, adding yeast nutrients to the solution in regulated amounts during the propagating period and periodically restoring the starting conditions in the fermenter by withdrawing a portion of the yeast containing liquid and diluting the remainder thereof.

7. A process of manufacturing yeast, which comprises starting propagation with a suspension of seed yeast in a fermenter partially filled with a dilute nutrient solution, aerating the solution, adding yeast nutrients to the solution in regulated amounts during the propagating period, periodically concurrently diminishing the addition of nutrients, lowering the aeration and withdrawing a portion of the yeast containing liquid, and thereafter approximately restoring the starting conditions in the fermenter by diluting the remainder of the yeast containing liquid with water and continuing the propagation therein with increased aeration and addition of nutrients.

8. A process of manufacturing yeast, which comprises starting propagation with a suspension of seed yeast in a fermenter partially filled with a dilute nutrient solution, aerating the solution, adding yeast nutrients to the solution in regulated amounts during the propagating period and periodically restoring certain of the starting conditions in the fermenter by withdrawing a portion of the yeast containing liquid and diluting the remainder thereof, the temperature of the liquid in which the seed yeast is suspended and the temperature maintained during the first few hours only of the period of propagation being in the neighborhood of 38 ° C.

9. A process of manufacturing yeast, which comprises starting propagation with a suspension of seed yeast in a fermenter partially filled with a dilute nutrient solution, aerating the solution, adding yeast nutrients to the solution in regulated amounts during the propagating period, the temperature of the nutrient solution in the fermenter being maintained for the first few hours only above 30° C., periodically concurrently diminishing the addition of nutrients, lowering the aeration and withdrawing a portion of the yeast containing liquid, and thereafter approximately restoring certain of the starting conditions in the fermenter by diluting the remainder of the yeast containing liquid with water and continuing the propagation therein with increased aeration and addition of nutrients.

10. A process of manufacturing yeast, which comprises preparing a relatively concentrated yeast nutrient solution, initiating propagation of yeast in a fermenter containing a diluted portion of the nutrient solution, aerating the solution, adding the remainder of the concentrated solution to the diluted portion in regulated amounts during the period of propagation, periodically withdrawing a portion of the yeast containing liquid, diluting the residue thereof and then continuing the propagation with the addition of nutrients.

11. A process of manufacturing yeast, which comprises preparing a relatively concentrated yeast nutrient solution, initiating propagation of yeast in a fermenter containing a diluted portion of the nutrient solution, aerating the solution, adding the remainder of the concentrated solution to the diluted portion in regulated amounts during the propagation period, periodically concurrently diminishing the addition of the concentrated solution, lowering the aeration, and withdrawing a portion of the yeast containing liquid, and thereafter diluting the residue thereof and continuing the propagation therein with increased aeration and addition of the concentrated nutrient solution.

12. A process of manufacturing yeast, which comprises preparing a relatively concentrated yeast nutrient solution, initiating propagation of yeast in a fermenter containing a diluted portion of the nutrient solution, aerating the solution, adding to the solution during the period of propagation and in

regulated amounts, yeast nutrients including the remainder of the concentrated solution, periodically concurrently diminishing the addition of nutrients, lowering the aeration, and withdrawing a portion of the yeast containing liquid, and thereafter diluting the residue thereof and continuing the propagation therein with resumption of higher aeration and addition of nutrients.

13. A process of manufacturing yeast, which comprises preparing a yeast nutrient solution including substantial amounts of sugar material and yeast-nutrient salts, initiating propagation of yeast in a fermenter containing a diluted portion of the nutrient solution, aerating the solution, adding to the diluted solution during the period of propagation and in regulated amounts the remainder of the concentrated solution together with other yeast nutrients, periodically withdrawing a portion of yeast containing liquid, diluting the residue thereof and then continuing the propagation therein with increased aeration and addition of nutrients.

14. A process of manufacturing yeast, which comprises preparing a yeast-nutrient solution including substantial amounts of sugar material and yeast nutrient salts, initiating propagation of yeast in a fermenter containing a diluted portion of the nutrient solution, aerating the solution, adding to the diluted solution during the period of propagation and in regulated amounts the remainder of the concentrated solution together with other yeast nutrients including an antacid substance, periodically withdrawing a portion of the yeast containing liquid, diluting the residue thereof and then continuing the propagation therein of increased aeration and addition of nutrients.

15. A process of manufacturing yeast, which comprises preparing a yeast nutrient solution including substantial amounts of sugar material and yeast-nutrient salts, initiating propagation of yeast in a fermenter containing a diluted portion of the nutrient solution, aerating the solution, adding to the diluted solution during the period of propagation and in regulated amounts the remainder of the concentrated solution together with other yeast nutrients including an antacid substance, periodically withdrawing a portion of the yeast containing liquid, diluting the residue thereof and then continuing the propagation therein with increased aeration and addition of nutrients, the temperature of the diluted nutrient solution in which the propagation is initiated and the temperature maintained during the first few hours only of a period of propagation being above 30° C.

16. A process of manufacturing yeast, which comprises preparing a yeast nutrient solution including substantial amounts of sugar material and yeast-nutrient inorganic salts, initiating propagation of yeast in a fermenter containing a diluted portion of the nutrient solution, aerating the solution, adding to the diluted solution during the period of propagation and in regulated amounts the remainder of the concentrated solution together with other yeast nutrients including an antacid substance, periodically concurrently diminishing the addition of nutrients, lowering the aeration and withdrawing a portion of the yeast containing liquid, and thereafter diluting the residue thereof and continuing the propagation therein with increased aeration and addition of nutrients.

17. A process of manufacturing yeast, which comprises preparing a yeast nutrient solution including substantial amounts of molasses and yeast-nutrient inorganic salts, initiating propagation of yeast in a fermenter containing a diluted portion of the nutrient solution, aerating the solution, adding to the diluted solution during the period of propagation and in regulated amounts the remainder of the concentrated solution together with other yeast nutrients including an antacid substance, periodically withdrawing a portion of the yeast containing liquid, diluting the residue thereof and then continuing the propagation therein with increased aeration and addition of nutrients.

18. A process of manufacturing yeast, which comprises preparing a yeast nutrient solution including substantial amounts of molasses and yeast-nutrient inorganic salts, initiating propagation of yeast in a fermenter containing a diluted portion of the nutrient solution, aerating the solution, adding to the diluted solution during the period of propagation and in regulated amounts the remainder of the concentrated solution together with other yeast nutrients including an antacid substance containing yeast-assimilable nitrogen, periodically withdrawing a portion of the yeast containing liquid, diluting the residue thereof and then continuing the propagation therein with increased aeration and addition of nutrients.

19. A process of manufacturing yeast, which comprises preparing a yeast nutrient solution including substantial amounts of molasses and yeast-nutrient inorganic salts, initiating propagation of yeast in a fermenter containing a diluted portion of the nutrient solution, aerating the solution, adding to the diluted solution during the period of propagation and in regulated amounts the remainder of the concentrated solution together with other yeast nutrients including an antacid substance, periodically withdrawing a portion of the yeast containing liquid, diluting the residue thereof and then continuing the propagation therein with increased aeration and addition of nutrients, the temperature of the diluted nu-

trient solution in which the propagation is initiated and the temperature maintained during the first few hours only of a period of propagation being above 30° C.

20. A process of manufacturing yeast, which comprises preparing a yeast nutrient solution including substantial amounts of molasses and yeast-nutrient inorganic salts, initiating propagation of yeast in a fermenter containing a diluted portion of the nutrient solution, aerating the solution, adding to the diluted solution during the period of propagation and in regulated amounts the remainder of the concentrated solution together with other yeast nutrients including an antacid substance containing yeast-assimilable nitrogen, periodically withdrawing a portion of the yeast containing liquid, diluting the residue thereof and then continuing the propagation therein with increased aeration and addition of nutrients, the temperature of the diluted nutrient solution in which the propagation is initiated and the temperature maintained during the first few hours only of a period of propagation being above 30° C.

21. An improved method of carrying on a continuous-addition process of manufacturing yeast by propagation with aeration, which includes periodically interrupting the addition of yeast nutrients, withdrawing a portion of the yeast containing solution, diluting the residue thereof and then continuing the propagation therein with the addition of yeast nutrients.

22. An improved method of carrying on a continuous-addition process of manufacturing yeast by propagation with aeration over a period of at least eight hours, which includes initiating propagation of yeast at a temperature above 30° C., and during the first few hours of the propagation allowing the temperature to fall to below 30° C.

23. An improved method of carrying on a continuous-addition process of manufacturing yeast by propagation with aeration, which includes periodically interrupting the addition of yeast nutrients, withdrawing a portion of the yeast containing liquid, diluting the residue thereof with water and then continuing the propagation therein, the temperature of the yeast containing solution during the first few hours only of at least one of the periodic stages being above 30° C.

24. An improved method of carrying on a continuous-addition process of manufacturing yeast by propagation with aeration, which includes the steps of periodically concurrently diminishing the addition of yeast nutrients, lowering the aeration and withdrawing a portion of the yeast containing liquid, and thereafter diluting the residue thereof and continuing the propagation therein with increased aeration and addition of yeast nutrients.

25. An improved method of carrying on a continuous-addition process of manufacturing yeast by propagation with aeration, which includes the steps of periodically concurrently diminishing the addition of yeast nutrients, lowering the aeration and withdrawing a portion of the yeast containing liquid, and thereafter diluting the residue thereof, and continuing the propagation therein with increased aeration and addition of nutrients, the temperature of the yeast containing solution during the first few hours only of at least one of the periodic stages being above 30° C.

26. A process of manufacturing yeast by propagation with aeration, which comprises suspending seed yeast in a non-toxic liquid, aerating the liquid, adding yeast nutrients to the liquid in regulated amounts during the propagating period, periodically concurrently diminishing the addition of nutrients, lowering the aeration and withdrawing a portion of the yeast containing liquid, and thereafter diluting the residue thereof, removing yeast from the withdrawn yeast-containing liquid, returning a portion of such yeast to the diluted residue and continuing the propagation therein with increased aeration and addition of nutrients.

27. A process of manufacturing yeast by propagation with aeration, which comprises suspending seed yeast in a non-toxic aqueous liquid, aerating the liquid, adding yeast nutrients to the liquid in regulated amounts during the propagating period, periodically concurrently diminishing the addition of nutrients, lowering the aeration and withdrawing a portion of the yeast containing liquid, and thereafter diluting the residue thereof, continuing the propagation therein with increased aeration and addition of nutrients, and during the period of propagation neutralizing the deleterious excess of acidity by the controlled addition of a suitable antacid substance.

28. An improved method of carrying on a continuous-addition process of manufacturing yeast by propagation with aeration over a period of about eight hours, which includes initiating the propagation of yeast with aeration in a small portion of dilute nutrient solution at a temperature in the neighborhood of 33° C., gradually adding a yeast-nutrient solution thereto, and after a few hours slowly lowering the temperature to below 30° C.

29. An improved method of carrying on a continuous-addition process of manufacturing yeast by propagation with aeration over a period of about eight hours, which includes initiating the propagation of yeast with aeration in a small portion of dilute nutrient solution at a temperature in the neighborhood of 33° C., gradually adding a yeast-nutrient solution thereto, and after a

few hours gradually lowering the temperature to below 30° C. and holding it there for the remainder of the propagating period

30. An improved method of carrying on a continuous-addition process of manufacturing yeast by propagation with aeration over a period of about eight hours, which includes preparing a yeast-nutrient solution including molasses as a sugar material, initiating the propagation of yeast with aeration in a small portion of the nutrient solution, diluted with water and at a temperature in the neighborhood of 33° C., gradually adding the prepared nutrient solution during the eight-hour period, and a few hours after the initiation of the propagation gradually lowering the temperature to below 30° C.

In testimony whereof we affix our signatures.

ROBERT L. CORBY.
WILHELM H. F. BÜHRIG

PLAINTIFF'S EXHIBIT P-6.

Certified Copy of File Wrapper and Contents of United States Patent No. 1,449,103 to Friedrich Hayduck, Assignor to The Fleischmann Company, Dated March 20, 1923.

(Physical Exhibit.)

PLAINTIFF'S EXHIBIT P-7.

Certified Copy of File Wrapper and Contents of United States Patent No. 1,449,105 to Friedrich Hayduck, Assignor to The Fleischmann Company, Dated March 20, 1923.

(Physical Exhibit.)

PLAINTIFF'S EXHIBIT NO. 8.

Certified Copy of File Wrapper and Contents of United States Patent No. 1,449,106 to Friedrich Hayduck, Assignor to The Fleischmann Company, Dated March 20, 1923.

(Physical Exhibit.)

PLAINTIFF'S EXHIBIT P-9.

Certified Copy of File Wrapper and Contents of United States Patent No. 1,449,109 to Friedrich Hayduck, Assignor to The Fleischmann Company, Dated March 20, 1923.

(Physical Exhibit.)

PLAINTIFF'S EXHIBIT P-10.

Certified Copy of File Wrapper and Contents of United States Patent No. 1,673,735 to Robert L. Corby and Wilhelm H. F. Buhrig, Assignor to The Fleischmann Company, Dated June 12, 1928.

(Physical Exhibit.)

PLAINTIFF'S EXHIBIT P-11.

UNITED STATES DISTRICT COURT
FOR THE DISTRICT OF NEW JERSEY

STANDARD BRANDS INCORPORATED, Plaintiff, —VS— NATIONAL GRAIN YEAST CORPORATION, Defendant.	In Equity No. E-4552 On United States Letters Patent Nos. 1,449,103, 1,449,105, 1,449,106, 1,449,109, and 1,673,735

CONSOLIDATION OF PLAINTIFF'S INTERROGATORIES AND DEFENDANT'S ANSWERS.

Interrogatory I(a). Has the defendant, within six years prior to December 30, 1932, practiced a process of manufacturing bakers' yeast with aeration using molasses as all or part of the sugar material?

Answer: Yes.

Int. I(b). In such process what sugar material (if any) other than molasses was used, and in what proportion to the total sugar material was the molasses?

Answer: Extractives of saccharified malt and other starch-bearing materials subject to saccharification in varying proportions depending upon the quality of the malt, the amount of available nutrient which can be utilized by the yeast, the price and composition of the raw materials and the judgment of the operator. The amount of molasses has varied between 85% and 90% of the total sugar material.

Int. I(c). If the answer to Interrogatory I(a) is in the affirmative, has defendant used ammonium phosphate in said process?

Answer: Yes, as a source of phosphorus in accordance with the common practices and teachings of the prior art.

Int. I(d). If the answer to Interrogatory I(a) is in the affirmative, has defendant used ammonium sulphate in said process?

Answer: Yes, as a source of nitrogen in accordance with the common practices and teachings of the prior art.

Int. I(e). If the answer to Interrogatory I(a) is in the affirmative, has defendant added a neutralizing agent to the fermenting liquid during the yeast propagation period?

Answer: Yes, at times—no, at other times, depending upon the variables given in answer to I(b).

Int. I(f). If the answer to Interrogatory I(e) is in the affirmative, has defendant added aqua ammonia to the fermenting liquid during the yeast propagation period?

Answer: Not as regular practice, only infrequently in emergencies caused by variations in raw materials which produce variations in the fermenting liquid.

Int. I(h). If the answer to Interrogatory I(e) is in the affirmative, has defendant added di-ammonium phosphate to the fermenting liquid during the yeast propagation period?

Answer: Defendant has added di-ammonium phosphate to the fermenting liquid during the yeast propagation period.

Int. I(i). In case the defendant has answered affirmatively any of the Interrogatories numbered I(a) to I(h) inclusive, which of the steps so admitted were used in the same run of yeast manufactured at any time during the term stated?

Answer: Defendant has used at times in the same run I(a), I(b), I(c), I(d), I(h) and I(e) at times as stated in answers I(b) and I(e).

Int. II. If the answer to Interrogatory I(e) is in the affirmative, then as to such process state the range of acidity maintained, and the range of acidity desired, during each hourly period of the propagation process.

Answer: The degree of acidity is and was maintained below the point of absolute toxicity. The acidity was measured by any of the well-known methods and with a knowledge of the degree or point of toxicity dependent upon the kind of acid used or released from an ammonium compound during fermentation. The degree of toxicity varies hourly depending upon the culture and the kinds of acid, and the concentration of the wort because the more concentrated the wort the greater the buffering action.

Int. III. Has the defendant, within six years prior to December 30, 1932, practiced a process of manufacturing bakers' yeast by initiating the propagation of yeast with aeration in a dilute yeast-nutrient solution (such as about 2° Balling or less), and, during a continuing period of further propagation, adding thereto further quantities of yeast-nutrients in solution?

Answer: Yes, within the Balling range of the prior art and the procedure of the additions of the prior art.

Int. IV. If the answer to Interrogatory III is in the affirmative, has the defendant prepared a yeast-nutrient solution and initiated such propagation in a dilute portion thereof, and during a period of further propagation added thereto a more concentrated nutrient solution (such as the remainder of the prepared yeast-nutrient solution)?

Answer: Yes, in accordance with the prior art.

Int. V. If the answer to Interrogatories III and IV, or either of them, is in the affirmative, did the dilute

nutrient solution, in which the propagation was initiated, contain some quantity of all essential yeast-nutrients?

Answer: Yes, in accordance with the prior art.

Int. VI. If the answer to either Interrogatory III or IV is in the affirmative, state what yeast-nutrients were added and when, how, and in what amounts such additions were made.

Answer: Yeast nutrients such as are available from molasses, barley malt, malt sprouts and ammonium compounds which yield nitrogen and phosphorus and other mineral nutrients essential to yeast growth, were used at such times and in such amounts as are necessary to sustain yeast growth.

Int. VII. Has the defendant within six years prior to December 30, 1932, practiced a process of manufacturing bakers' yeast in which the propagation of yeast was initiated in a dilute nutrient solution containing an ammonium salt of an inorganic acid, aerating the solution and adding thereto, during the propagation period, yeast-nutrient materials including a substance having an alkaline reaction and containing yeast-assimilable nitrogen?

Answer: Yes, according to the prior art.

Int. X. If the answer to the foregoing Interrogatories or any of them is in the affirmative, then as to each so affirmed, the defendant shall furnish the plaintiff with mash records and informative descriptions of two runs typifying such process, giving complete data as follows:

(a) The steps, materials and amounts thereof used in the mashing operations and in the preparation of the nutrient solution and during the propagation.

(b) The time of each operation.

(c) The temperatures, densities, acidities (including a description of the scale of acidity and the method

of determining the same) of each solution used and of the propagating liquid at hourly or half-hourly periods during the manufacture.

(d) The amount of seed yeast used.

(e) The yield of yeast obtained (stating the moisture content).

(f) The weight of each of the raw materials used.

(g) Typical samples of each of the raw materials used in the preparation of the nutrient solution, sufficient in quantity for analysis by plaintiff.

(h) The concentration and total volume of the extract and wash waters obtained from mashing the cereal materials.

(i) The Balling of the original molasses, if such be used.

(j) The method, if any, of the treatment of molasses prior to use for yeast propagation.

(k) The Balling and volume of the solution obtained from the molasses.

(l) The concentration and volume of the liquid initially in the fermenter at the beginning of the propagating period.

(m) The total volume of liquid at the end of the propagating period, together with a statement of how much, if any, has been added or withdrawn during such period.

(n) The amounts of nutrient material added during the propagating period, and the times and amounts of each such addition and, if solid, whether in water solution.

(o) The amounts of any liquid withdrawn during the propagating period, and full information as to what was done therewith.

(p) The Balling of the propagating liquid from hour to hour, including the final Balling of the yeast-containing solution at the end of the propagating period.

(q) The amount of total nitrogen, ammonia nitrogen, and alcohol, left in the beer after the yeast has been separated therefrom.

(r) An analysis typical of the seed yeast and final yeast product.

(s) The amount of aeration used.

Answer: Defendant's process involves a prolonged lactic acid souring of a combined grain and molasses mash which supplies a large amount of natural organic nitrogen liberated from the basic materials as a result of excessive protein hydrolysis. There also results the formation of a large amount of lactic acid, a desirable natural organic acid, which is combined as it is formed, with aqua ammonia to form ammonium lactate, which accumulates in the mash. Ammonium acid phosphate and di-ammonium phosphate are employed to supply the needed phosphorous and ammonium sulfate is added to supply additional nitrogen in a form to maintain a favorable acidity of the wort. Occasionally additions of mineral acid are required for this purpose.

Records of a typical run at about the time the suit was filed are attached.

Basic materials:—

Beet molasses	23,000 pounds
Malt	3,600 pounds
Malt sprouts	3,900 pounds

GRAIN MASH:

The ground malt and malt sprouts are mixed with cold water in the mash tank and are mashed by the

usual well known method, for peptonization of the proteins and conversion of the starch.

Peptonization is accomplished by gradually heating the mixture to 134° F. and holding at this temperature for one-half hour. Next the mash is heated to 146° F. and held at this temperature for about two hours or until all the starch has been converted into sugar. Next the mash is cooled to 128° F. and stocked with a pure culture of Lactobacillus Delbrucki. This grain mash (total volume 6,500 gallons) is allowed to sour for twenty-four hours and during this time a total of 204 pounds of acqua ammonia is added to the mash in six portions to combine with the lactic acid as it is being formed.

Molasses Mash:

In the meantime, the molasses is being prepared in a separate tank. The 23,000 pounds of molasses (approximately 2000 gallons) is diluted with 3000 gallons of water and acidified with 144 pounds of sulphuric acid. The mixture is heated with steam and during the heating process, there is added 96 pounds of monocalcium phosphate. Heating is continued to the boiling point and then the whole mixture is allowed to stand over night.

Grain and Molasses Mixed:

The molasses mash which has been standing over night is now decanted off and pumped to the souring tank on top of the grain mash. More aqua ammonia is added from time to time to combine with the lactic acid as formed, until a total of from 350 to 400 pounds of ammonia has been added during the two days of souring. Next the mash is sterilized by heating with steam to 160° F. On the morning of the third day the sterile mash is pumped to the filter tub where the liquid extract is separated from the insoluble grain residues.

The filtered mash is used for making both seed yeast and commercial yeast and is divided into two parts, between one-fifth and one-sixth being used for the seed.

The portion for seed yeast is taken from the first run heavy extract, coming from the filter tub and is set aside in a separate tank. This is used for making one large fermenter of Seed Yeast.

Of the balance of the mash to be used for commercial yeast a sufficient part is set aside in another separate tank, for setting the commercial fermenters.

The balance of the wort or extract is pumped into a large wooden storage tank of 16,500 gallons' capacity. The insoluble grains in the filter tub are repeatedly sparged with hot water in order to wash out all soluble extract and * these spargings are used to dilute the wort to be added to the fermenters during yeast propagation. The dilute extract or spargings are added until the tank is full. This gives approximately 16,500 gallons of feed wort of approximately 12% Balling gravity.

The feed wort is well agitated with air to secure a mixture of uniform gravity and 257 pounds of ammonium sulphate are added to supply additional nitrogen as yeast nutrient. The balance of the inorganic salts used, including 250 pounds ammonium sulphate, 100 pounds mono-ammonium phosphate, and 350 pounds di-ammonium phosphate are added directly to the fermenters during propagation to supply nitrogen and phosphate as required by the yeast growth.

Four wooden fermenters of approximately 15,000 gallons' capacity, and two metal fermenters of approximately double that capacity are employed for the portion of the above described mash used for producing

*Note: Counsel agree that this should read "a part of these" spargings.

the commercial yeast. The four wooden fermenters are "set" by charging them with about approximately 9600 gallons of dilute wort solution made up chiefly of spargings used in washing the mash in the filter tub after the concentrated wort has been drawn off. The deficiency, if any, is made up by concentrated wort from the storage tank, and water. The concentration of the initial solution after seeding is approximately 1.3 Balling.

Each fermenter is seeded with a seed yeast solution of approximately 750 pounds and 20 pounds of ammonium sulphate, and 50 pounds mono-ammonium phosphate are added at the start.

After about one hour the feed of the concentrated wort from the storage tank is started from three feed cocks at the rate of 75 gallons each per hour. The temperature is maintained throughout as near as possible at 28° C. After two hours the feed is increased to four feed cocks and after a further two hours to five feed cocks and continued for two hours and fifteen minutes.

At hourly intervals the wort is tested for concentration and acidity. The method employed for testing the acidity is the standard neutralization test carried out by taking 40 cc. of sample and titrating with one-tenth normal sodium hydroxide solution, using litmus paper as indicator. Occasionally, but not as regular practice, the acidity is determined by measuring the hydrogen ion concentration.

At approximately one and a half or two hour intervals di-ammonium phosphate and ammonium sulphate are added to the wort in varying quantities in the order of eight to twenty pounds to supply the needed nitrogen and phosphate.

After seven and one-half hours water is added to bring the total volume up to about 13,000 gallons and

one-half of the total solution in two of the wooden fermenters discharged into one of the iron fermenters.

The fermentation is then continued in all the fermenters to which the concentrated wort is fed and diammonium phosphate and ammonium sulphate are added as indicated on the attached "Process Tables" dated December 9, 1932.

From the fermenters the wort is delivered to the centrifugal separators.

The attached process tables dated April 27, 1931 show a typical run of a somewhat modified process as employed by the defendant during the year 1931. The mash was prepared according to the same procedure described above, with certain differences in the relative proportions of the ingredients, as indicated. A portion of the mash was set aside for use in the seed fermenters and the balance after setting the fermenters was used as the feed wort.*

All the aqua ammonia was added to the feed wort to form ammonium lactate and all the ammonium phosphate and ammonium sulphate (320 pounds mono-ammonium phosphate and 162 pounds sulphate) was added directly to the fermenters, the **balance of ammonium sulphate being used in the seed fermenters.

In the process of which the April 27 records are typical, all the ammonium phosphate is added initially to the fermenter while the ammonium sulphate was added in part initially and with later small additions, as indicated on the tables. Also, as indicated, the acidity toward the end of the fermentation period was raised by the addition of one or two liters of acid. Dur-

*Note: Counsel agree that this sentence is incorrect in that no seed yeast was made from this mash and all of the mash materials were used in making the commercial yeast.

**Note: See preceding Footnote. Counsel agree that the balance of the ammonium sulphate 190 lbs. was added to the reserve wort, 50 lbs to that in the SCT tank, 100 lbs. to the LCT tank and 40 lbs. to the IP tank.

ing the operation of this process complete fermentation takes place in a single tank.

The foregoing, together with the mash records, contains all the information asked for under Interrogatory X except the following:

X(d). The amount of seed yeast varies between 9% and 12% of pressed yeast (70% moisture) on the weight of the raw materials.

X(e). The yield of yeast varies between 60% and 70% pressed yeast (70% moisture) on the weight of the raw materials.

X(g). Defendant has not in its possession now samples of any of the raw materials used by it prior to the filing of the Bill of Complaint herein. The materials used by defendant are standard commodities purchased in the open market. Defendant will on request furnish plaintiff with samples of materials which it is now using and which it believes to be of the same character as used before the Bill of Complaint was filed, except for the unavoidable variations in organic materials, particularly molasses.

X(i). Defendant has no figures as to the Balling of the original molasses.

X(k). The defendant has no figures except as given above of the Balling and volume of the solution obtained from the molasses.

X(q). Defendant has made no analysis of the beer after the yeast is separated therefrom. Defendant made no analysis of the seed yeast or the final yeast product except for protein content which averages over 50%.

X(s). Defendant has made no accurate measure of the amount of aeration used, but it is roughly from

800 to 1200 cubic feet per minute per 15,000 gallon fermenter.

(NOTE: On the Mash Records:

A indicates mono ammonium phosphate
B indicates aqua ammonia
C indicates ammonium sulphate
D indicates di-ammonium phosphate
C.P. indicates calcium phosphate)

PLAINTIFF'S EXHIBIT P-12.

UNITED STATES DISTRICT COURT
FOR THE DISTRICT OF NEW JERSEY

STANDARD BRANDS INCORPORATED, Plaintiff, —VS— NATIONAL GRAIN YEAST CORPORATION, Defendant.	In Equity No. E-4552 On United States Letters Patent Nos. 1,449,103, 1,449,105, 1,449,106, 1,449,109, and 1,673,735

SUPPLEMENTAL FACTS OF DEFENDANT'S OPERATIONS GIVEN BY DEFENDANT'S COUNSEL.

In addition to the information given by defendant in its answers to plaintiff's interrogatories as to flow sheets M177-M180, dated April 27, 1931, and flow sheets 3539-3544, dated December 9, 1932, defendant's counsel has supplied plaintiff's counsel with a mash sheet of September 12, 1931 and flow sheets therefor Nos. M559-M561, dated September 15, 1931, together with the following supplemental information relating to the aforesaid processes.

1. As to Mash and Flow Sheets M 177-M 180

(a) The figure 467 represents the amount of ammonia left in the drum from the preceding mash, and out of which the ammonia for the mash in question is deducted. It is merely an inventory figure.

(b) "18T" indicates 18 pounds of ammonium phosphate used in after treatment of yeast.

(c) "S. C. T." indicates small copper tank; "L.C.T." indicates large copper tank; "I.P." indicates iron pre-fermenter.

(d) "65" indicates 65 pounds of calcium phosphate.

(e) The figures circled indicate the number of cocks open on the feed wort line at the times indicated on the sheet.

(f) The word "Separating" indicates the appearance of the yeast, that is, that it had begun to show a flocculent appearance.

(g) The volume of reserve solution in "S.C.T." was 2,500 gallons, in "L.C.T." 5,000 gallons and in "I.P." 2,000 gallons, which were fed successively into the fermenters. The total volume of the solution initially in the fermenters was 37,000 gallons and at the end the total volume was 46,500 gallons.

2. Mash and Flow Sheets 3539-3544

(a) These figures indicate that the total 3900 pounds of malt sprouts was mashed in two separate portions of 1500 and 2400, respectively.

(b) The figure "5" indicates five inches of molasses, one inch being equal to 512 pounds, "4L" indicates four liters of sulphuric acid, "6#" indicates six pounds calcium phosphate.

(c) The notation indicates 2500 gallons of feed wort put in the small copper tank for seed yeast.

(d) There are no arrows on Flow Sheet 3539. There appear to be some on 3542 and 3544. Wher-

ever these arrows appear they indicate changes that were made after the sheet was written up. The sheets are prepared in advance of the run as instructions to the foreman in charge. The changes were made after the original write-up and they are indicated by arrows appearing on these sheets.

(e) "2 Pails" indicates two pails of ammonium sulfate solution added to the seed yeast. The solution is of the strength of 60 pounds ammonium sulfate in 50 gallons of solution.

3. As to the Mash Sheets and Flow Sheets M 559-M 561

The total volume of the solution initially in the commercial fermenters was 28,000 gallons and the final volume was 35,000 gallons.

4. In all the processes typified by the above mentioned mash sheets, defendant has not analysed the spent wort for alcohol, has not recovered any alcohol.

Counsel for Defendant agrees that the above Supplemental Facts are correct.

PLAINTIFF'S EXHIBIT P-13.

Original Copy of Defendant's Mash Sheet of September 12 to 15, 1931.

Mash Saturday Sept 12th
For Tuesday Sept 15th
2 - 2nd Gen Seed + 3 conc 7's (wood)

Materials

Malt - 2600#
Sprouts - 3000#
Molasses - 16,700#

{ 2580# in mash tub
{ 500# in sour tank

Molasses Mash in New Tank

{ 32" Molasses
{ 26 L-acid
{ 80# Ca-Phosphate

Distribution of Materials

42% for Seed (2-Iron 7's) 58% for Conc (3 wood 7's)

140 - AP - 225+107 = 741
230 - Am - 240
224 - A.S - 258
34 - C.P - 46

Feed Wort - SCT - 17% B for Seed
L.C.T - 20% for Seed 80% for Conc
I.P. - 12% B - all for Conc. Yeast

Run to Sour tank - Monday morning

Sour Mash

11 P.M. - 11 - cc
2 A.M. - 23 - cc
6 A.M. - 34 - cc

11 P.M. - 28 - cc
6 A.M. - 28 cc

For Tuesday morning Heat to 170°F

Type new 9/15/31

NATIONAL GRAIN YEAST CORPORATION

Seed 138 Manufacturing Process—Table

Mash No. 26-A Date Sept 15, 1931

1st 7

RAW MATERIALS

Malt	1075	lbs.	
Sprouts	1250	lbs.	L.C.T 20% for Seed
Corn		lbs.	
		lbs.	
Molasses	7000	lbs.	
		lbs.	
Am. Phosphate	140 / 100	lbs.	+ 40
Ammonia	56 /	lbs.	S.C.T for Seed
Am. Sulphate	448 / 368	lbs.	+ 40 → +(20 from L.C.T.)
C.P.	35	lbs.	

Acid Added

For Neutralization

During Fermentation

In Liquid Yeast

Am 744 -254 487

Total Materials 40 + 16 lbs.

Molasses

Total gallons molasses and water

Sterilizing Temperature

Sterilizing Time 17.0% Ash From M. to M.

Time start running in fermenter

Gravity of first extract SCT {17.1 / 22-} 20

Acidity of first extract

Gallons in Fermenter

Grain Mash

Start Filtering 7:45 A M.

Finish Filtering 3 20 P. M.

From M. to M.

Stocked with 5/1 448# Lbs. of No. 138 Seed 1st Gen

Yield of Yeast, Net lbs. % Yield

Fermenting strength of yeast

Set at 2.5 Process of Fermentation (A) (C)

	10 TIME Hours	% BALLG	°C	ACIDITY			
1	2	2.50	27	2.8	50 / 100 / AIR		Seed
2	3	2.25	27	3.1			
3	4	1.90	275	4.0			2 30 (2)
4	5	1.55	27	4.5	2 Pails Blower		
5	6	1.35	265	4.8	3 "		
6	7	1.45	27	3.5	3 "	6 00 more air	6 01 (3)
7	8	1.60	N27	3.8	2.5 " 3 "		
8	9	1.70	27	3.0	1.5 " 4 "		9 01 (4)
9	10	1.85	275	3.0	3 "		
10	11	2.05	27	3.2	3 "		10 30 (2)
11	12	2.1	27	3.0			
12	1	2.3	27	3.5	18#		11 30 finish
13		2.4	27	4.1			

Shipped 24

~~Put~~ 2 Liters Acid in each tank

2nd Gen Seed - Use #2 tank

Clean Everything Good

For Seed

X

Time run to separators, From 10 01 M. to 2 15 M. No. of separators

Name

Lion Fermenter No. I

NATIONAL GRAIN YEAST CORPORATION

Seed 138 Manufacturing Process—Table

Mash No. 20-A / 2nd 7 Date Sept 15, 1931

RAW MATERIALS

Malt _____ lbs.
Sprouts _____ lbs.
Corn _____ lbs.
_____ lbs.
Molasses _____ lbs.
_____ lbs.
Am. Phosphate _____ lbs.
Ammonia _____ lbs.
Am. Sulphate _____ lbs.
_____ lbs.

Acid Added
For Neutralization _____
During Fermentation _____
In Liquid Yeast _____

Total Materials _____ lbs.

	Molasses			Grain Mash		
Total gallons molasses and water				Start Filtering		
Sterilizing Temperature				Finish Filtering		
Sterilizing Time	From	M. to	M.	From	M. to	M.
Time start running in fermenter						
Gravity of first extract						
Acidity of first extract						
Gallons in Fermenter						

Seeded with 8/4" = 448 Lbs. of No. 13 F Seed. 1st gen

Yield of Yeast, Net _____ lbs. _____ % Yield

Fermenting strength of yeast _____

Process of Fermentation (A) (C)

2.5

	TIME Hours	% BALLG.	°C.	ACIDITY	(A)	(C)	
24		2.50	27	2.8	50 ✓	100 ✓	Feed
1	3	2.20	27	3.2			4 30 (2)
2	4	1.85	27	3.7			
3	5	1.50	27	4.1		2 Pails	7 30 (3)
4	6	1.45	27	4.3		3 "	
5	7	1.50	27	3.6		3 "	
6	8	1.60	27	3.0	2 L.	3 "	10 30 (4)
7	9	1.80	27	2.8	1 L.	4 "	
8	10	1.95	27	3.4		3 "	12 00 (5)
9	11	2.0	27	2.8		3 ✓	
10	12	2.15	27	3.3			12 45 finish
11	1	2.3	27	4.3		18 4	
12	2 15	2.4	27.5	5.0		✓	
13							
14							
15							24 new note
16							
17							
18							
19							
20							

Time run to separators, From 2 15 to 3 30 M. No. of separators

Name

Inn Fermenter No. 2

NATIONAL GRAIN YEAST CORPORATION

Mash No. M-559 Manufacturing Process—Table Date Sept 15, 1931

RAW MATERIALS

Malt	1525 lbs.	Acid Added	114 a 94%
Sprouts	1750 lbs.	For Neutralization	
Corn	lbs.	During Fermentation	
	lbs.	In Liquid Yeast	
Molasses	9700 lbs.		
	lbs.		
Am. Phosphate	225 + 10T lbs.		
240 Ammonia	94 + 19 lbs.		
258 Am. Sulphate	333 193 lbs.		
Cu. P.	45 lbs.		

$\frac{LCT}{80 + (20 \text{ Acid})} + \frac{IP}{40}$ = 120 # infect for Conc.

100 #

Total Materials 64 + 38 lbs.

	Molasses	Grain Mash
Total gallons molasses and water	80% Cu. 4	
Sterilizing Temperature	20% Acid	
Sterilizing Time	From M. to M.	From M. to M.
Time start running in fermenter	(11.3)	
Gravity of first current	LCT 17.6	IP 11.93
Acidity of first current	23	18
Gallons in Fermenter	5000	5000

Stocked with 375 Lbs. of No. 137. K- Bacillus B

Yield of Yeast, Net lbs. % Yield

Remaining strength of yeast

1.4% B

Process of Fermentation (A) (C)

	TIME Hours	% BALLG	°C	ACIDITY			
1	3 30	1.40	27.5	1.5	75	90	11R
2	4 30	1.20	28	1.8			
3	5 30	1.05	28	2.4			
4	6 30	1.00	28	2.9		14	
5	7 30	1.10	28	3.2			
6	8 30	1.20	28	2.8		14	
7	9 30	1.35	28	2.6			
8	10 30	1.45	28	2.8		14	
9	11 30	1.5	28.5	2.8			
10	12 30	1.6	28	3.1	B 12 oz		
11	1 30	1.7	28.5	3.0			
12	2 30	1.85	28.5	3.5	11R		
13	3 30	2.0	29	3.0			
14	4 30	2.15	29	3.4	10 oz		
15	5 30	2.3	29	3.0			
16	6 30	2.4	28.5	3.3		62	
17		2.5	27.5	3.5			
18							
19							
20							

Compressor—running on 3 Tanks

Feed

3 30 (1)

5 30 (2)

7 30 (3)

2 oz (4)

5 00 finish O.K.

22½

31½

4 #
3 #
3 #

10 # A.B on wash tank

12-2 - Acid in " "

12-2. acid in wash tank.

X

These run to separators, From 6 30 M. to 7 30 M. No. of separators 5

Name

Wood Fermenter No. #I

NATIONAL GRAIN YEAST CORPORATION

Mash No. M-560 Manufacturing Process—Table Date Sept 15, 1931

RAW MATERIALS

Malt _____ lbs.
Sprouts _____ lbs.
Corn _____ lbs.
_____ lbs.
Molasses _____ lbs.
_____ lbs.
Am. Phosphate _____ lbs.
Ammonia _____ lbs.
Am. Sulphate _____ lbs.
_____ lbs.

Acid Added
For Neutralization _____
During Fermentation _____
In Liquid Yeast _____

Total Materials _____ lbs.

	Molasses			Grain Mash		
Total gallons molasses and water				Start Filtering		
Sterilizing Temperature				Finish Filtering		
Sterilizing Time						
Time start running in fermenter	From	M. to	M.	From	M. to	M.
Gravity of first extract						
Acidity of first extract						
Gallons in Fermenter						

Stocked with 375 Lbs. of No. 137 K. Bottom B

Yield of Yeast, Net _____ lbs. _____ % Yield

Fermenting strength of yeast _____

Process of Fermentation (A) (C)

	TIME Hours	% BALLG.	°C	ACIDITY			
3 30		1.40	28	1.5	75	20	Feed
1	4 30	1.15	28	1.8			
2	5 30	1.00	28	2.4			4 30 ①
3	6 30	1.00	28	2.8		14	
4	7 30	1.10	28	3.0			6 30 ②
5	8 30	1.25	28	2.9		14	
6	9 30	1.35	28	2.7			8 30 ③
7	10 30	1.45	28	2.8		14	
8	11 30	1.5	28	2.9		B	3 ④
9	12 30	1.6	28.5	3.4		12 =	
10	1 30	1.75	28.5	3.2			
11	2 30	1.9	29	3.5		12	6 ⑤
12	3 30	2.05	29	3.1			finish
13	4 30	2.2	29	2.8		11	9 K.
14	5 30	2.3	28.5	3.0			
15	6 30	2.4	28	3.3			~~32~~
16	7 31	2.45	28	3.6		75	31 1/2
17							
18							
19							X
20							

Time run to separators, From 7 31 M. to 8 30 M. No. of separators _____

Name _____

Wood Fermenter No. 2

NATIONAL GRAIN YEAST CORPORATION

Mash No. M-561 Manufacturing Process—Table Date Sept 15, 1931

RAW MATERIALS

Malt _____ lbs.
Sprouts _____ lbs.
Corn _____ lbs.
_____ lbs.
Molasses _____ lbs.
_____ lbs.
Am. Phosphate _____ lbs.
Ammonia _____ lbs.
Am. Sulphate _____ lbs.
_____ lbs.

Acid Added
For Neutralization _____
During Fermentation _____
In Liquid Yeast _____

Total Materials _____ lbs.

	Molasses			Grain Mash		
Total gallons molasses and water						
Sterilizing Temperature						
Sterilizing Time				Start Filtering		
Time start running in fermenter	From	M. to	M.	Finish Filtering From	M. to	M.
Gravity of first extract						
Acidity of first extract						
Gallons in Fermenter						

Stocked with 375 Lbs. of No. 137 1 Ger B

Yield of Yeast, Net _____ lbs. _____ % Yield

Fermenting strength of yeast _____

Process of Fermentation (A) (C)

	TIME Hours	% BALLG.	°C.	ACIDITY			
4 30							
1	5 30	1.40	27	1.5	75	20	Feed
		1.20	28	1.9			
2	6 30	1.00	28	2.3			5 30 ①
3	7 30	0.95	28	2.7		14	
4	8 30	1.10	28	2.9			7 30 ②
5	9 30	1.15	28	2.9		14	
6	10 30	1.3	28	2.6			9 30 ③
7	1 30	1.4	28.5	3.1		B 12	
8	12 30	1.5	29	3.1			4 00 ④
9	1 30	1.65	29	3.3		12	
10	2 30	1.8	28.5	3.0			7 00 finish O.K.
11	3 30	1.95	28	3.4		12	
12	4 30	2.05	28	3.1			
13	5 30	2.12	28	3.3		10	
14	6 30	2.3	28	3.1			
15	7 30	2.4	28	3.3		62	
16	8 30	2.4	27	3.3		45	31/2
17							
18							
19							
20							

Time run to separators, From 8 30 M. to 9 30 M. No. of separators _____

Name _____

Wood Fermenter No. 4

Mash to be made Saturday 4/25/31
For Monday 4/27/31
2 Iron 7's — Nos M-177-8
2 Wood 7's — Nos M-179-80

Materials

Malt - 2100
Sprouts - 2000+300
Molasses - 13,350

"26" Molasses
16 L - Acid
16 5-# Ca-Phos

Chemicals

A.P. - 320
Am - 340
A.S - 380

on 46 →

53% to Iron 7's
47% to Wood 7's

New Sour tub

Sour Mash Treatment

1st day Grain Mash			2nd day Molasses.		
Time	Test	Ammonia			
10a	12cc	10 Lc	3 PM	32 cc	16 Lc
-		.0.			

a. P. – 320
am – 340
a. S – 380

53% to Iron 7's
47% to Wood 7's

New Sour tub

Sour Mash Treatment

1st day Grain Mash			2nd day Molasses.		
Time	Test	Ammonia			
10a	12 cc	10 Lv	3:30 PM	32 cc	15 Lv
12n	18 cc	10 Lv	10a	34 cc	15 Lv
2p	20 cc	14 Lv	1a	32 cc	12 Lv
4p	22 cc	14 Lv			42 Lv
6p	24 cc	10 Lv	4a		86 lbs
		64 Lv	6a	30 cc	+128
		128 lbs			214#

Half to 1650 7
New Sour Tub
30 cc

PLAINTIFF'S EXHIBIT P-14.
Photostat of Defendant's Mash Sheet of April 25 to 27, 1931.

NATIONAL GRAIN YEAST CORPORATION

Mash No. M-177 Manufacturing Process—Table Date 4/27/31

RAW MATERIALS

Malt _____ lbs.
Sprouts _____ lbs.
Corn _____ lbs.
_____ lbs.
Molasses _____ lbs.
_____ lbs.
320 Am. Phosphate +19T ✓ lbs.
340 Ammonia (364) ✓ lbs.
380 Am. Sulphate (35 ✓) 162 lbs.
65 _____ lbs.

Total Materials _____ lbs.

Acid Added

For Neutralization _____

During Fermentation _____

In Liquor -467 (B) mash
-364 214 #
-103 +150 wtr
364 #

S&I 50 ✓ + LCT 100 + IP 40 ✓

Molasses

Total gallons molasses and water
Sterilizing Temperature
Sterilizing Time
Time start running in fermenter 11 00 A M
Gravity of first extract
Acidity of first extract
Gallons in Fermenter

From M. to M. (12.00)
VCT 13.6 / 21- 70 ✓
LCT 14.3 / 22- 40 ✓

Grain Mash

Start Filtering 8 00 A M.
Finish Filtering 4 00 P M.
From M. to M. (12.0)
IP 24.2 / 130 15 ✓

Stocked with 400 Lbs. of No. 117K Seed.

Yield of Yeast, Net _____ lbs. _____ % Yield

Fermenting strength of yeast _____

Process of Fermentation (A) (C)

12 =	TIME Hours	% BALLG.	°C	ACIDITY			
		1.00	28.5	1.5	85 ✓	30 ✓	Feed
1	1	0.90	28	1.7			1st (1)
2	2	0.85	28	2.1			
3	3	0.90	28	2.4			3rd (2)
4	4	1.00	28	2.6			
5	5	1.05	28	2.8	7 ✓		7.30 (3)
6	6	1.20	28	3.2			
7	7	1.35	28	4.2			10.30 (4)
8	8	1.5.	28	4.6			
9	9	1.6.	28	4.9	7 ✓		12.30 (5)
10	10	1.7.	28	4.8			
11	11	1.8.5	28	4.1			2nd finish
..	12	2.10.	28	3.6	.. 4		

Acidity of first extract 121-1 ✓ 122-1 ✓ 130 1 ✓

Gallons in Fermenter

Stocked with 400 Lbs. of No. 117K Seed.

Yield of Yeast, Net _____ lbs. _____ % Yield

Fermenting strength of yeast _____

Process of Fermentation (A) (C) ✓

	TIME Hours	BALLG.	°C	ACIDITY		
12 m		1.00	28.5	1.5	85 ✓	30
1	1	0.90	28	1.7		
2	2	0.85	28	2.1		
3	3	0.90	28	2.4		
4	4	1.00	28	2.6		
5	5	1.05	28	2.8	7 ✓	
6	6	1.20	28	3.2		
7	7	1.35	28	4.2		
8	8	1.5.	28	4.6		
9	9	1.6.	28	4.9	7 ✓	
10	10	1.7.	28	4.8		
11	11	1.8.5	28	4.1		
12	12	2.0.	28	3.6	44	
13	1	2.10	28	3.0		
14	2	2.2.	28	2.4	3 lt. acid	
15	3	2.2	28	3.1		
16	4 4 30	2.3.	28	2.3 2.8	3 lt. acid Separating	
17						
18	10 lbs Am Phos in 1st wash tank ✓					
19	8 lbs " " " 2nd " " ✓					
20	2 Liters Acid in each " " ✓					

Feed

1 oz (1)

3 oz (2)

7 30 (3)

10 30 (4)

12 30 (5)

2 oz finish ✓

35½ hrs

Time run to separators, From 4 30 M. to 5 50 M. No. of separators 5

Name

Press Rep. M-173-4 (2nd Tank) — Pressed — HHHII 23/4

" " M-175-6 (Big Tank) — Pressed — HHHII+1+1/4

IRON Fermenter No. I

NATIONAL GRAIN YEAST CORPORATION

Mash No. M-178 Manufacturing Process—Table Date 4/27/31

RAW MATERIALS

Malt _____ lbs.
Sprouts _____ lbs.
Corn _____ lbs.
_____ lbs.
Molasses _____ lbs.
_____ lbs.
Am. Phosphate _____ lbs.
Ammonia _____ lbs.
Am. Sulphate _____ lbs.
_____ lbs.

Total Materials _____ lbs.

Acid Added
For Neutralization _____
During Fermentation _____
In Liquid Yeast _____

Total gallons molasses and water
Sterilizing Temperature
Sterilizing Time
Time start running in fermenter
Gravity of first extract
Acidity of first extract
Gallons in Fermenter

Molasses
From M. to M.

Grain Mash
Start Filtering
Finish Filtering
From M. to M.

Stocked with 400 Lbs. of No. 117K Seed.

Yield of Yeast, Net _____ lbs. _____ % Yield

Fermenting strength of yeast _____

Process of Fermentation (A) (C)

	TIME	% BALLG.	°C	ACIDITY	15✓	30✓	Feed
1/30		1.00	28	1.5			
1	3 Hours	0.85	28	2.0			2 30 (1)
2	4	0.90	28	2.5			
3	5	0.95	28	3.0			4 30 (2)
4	6	1.05	28	3.0			
5	7	1.20	28	3.4	7✓		9 00 (3)
6	8	1.3	28	4.1			
7	9	1.4	28	4.5			12 00 (4)
8	10	1.5	28	4.6			
9	11	1.7	28.5	4.2	7✓		2 00 (5)
..	..	1.4	28	3.7			

Stocked with 400 Lbs. of No. 117K Seed.

Yield of Yeast, Net _____ lbs. _____ % Yield

Fermenting strength of yeast _____

Process of Fermentation

	TIME Hours	% BALLG.	°C	ACIDITY			Feed
1/30		1.00	28	1.5		85 ✓ 30 ✓	
1	3	0.85	28	2.0			2 30 ①
2	4	0.90	28	2.5			4 30 ②
3	5	0.95	28	3.0			
4	6	1.05	28	3.0			9 00 ③
5	7	1.20	28	3.4		7 ✓	
6	8	1.3	28	4.1			12 00 ④
7	9	1.4	28	4.5			
8	10	1.5	28	4.6			2 00 ⑤
9	11	1.7	28.5	4.2		7 ✓	
10	12	1.9	28	3.7			3 20
11	1	2.0	28	2.7		44	finish
12	2	2.1	28	2.0	3 lt. acid		
13	3	2.2	28.5	2.9			35 hrs
14	4	2.2	28	2.7			
15	5	2.3	28	2.3	3 lt. acid.		
16	5 50	2.3	28	2.8	Separating		
17							
18							
19							
20							

Time run to separators, From 5 50 M. to 7 10 M. No. of separators 5

Name _____

Iron Fermenter No. 2

RAW MATERIALS

Malt _____ lbs.
Sprouts _____ lbs.
Corn _____ lbs.
_____ lbs.
Molasses _____ lbs.
_____ lbs.
Am. Phosphate _____ lbs.
Ammonia _____ lbs.
Am. Sulphate _____ lbs.
_____ lbs.

Acid Added
For Neutralization _____
During Fermentation _____
In Liquid Yeast _____

Total Materials _____ lbs.

	Molasses	Grain Mash
Total gallons molasses and water		Start Filtering
Sterilizing Temperature		Finish Filtering
Sterilizing Time	From M. to M.	From M. to M.
Time start running in fermenter		
Gravity of first extract		
Acidity of first extract		
Gallons in Fermenter		

Stocked with 375 Lbs. of No. 117K Seed.

Yield of Yeast, Net _____ lbs. _____ % Yield

Fermenting strength of yeast _____

Process of Fermentation (A) (C)

3 00 TIME Hours	% BALLG. 1.00	°C 28.5	ACIDITY 1.5	75	25	Feed
1 4 00	0.90	28	1.8			4 00 (1)
2 5	0.85	28	2.5			
3 6	0.85	28	3.0			6 00 (2)
4 7	1.00	28	2.8			
5 8	1.1.	28	2.7		6	10 00 (3)
6 9	1.2.5	28	2.8			
7 10	1.4.	28	3.0			2 30 (4)
8 11	1.5.	28	3.2			
9 12	1.6.	28	3.3		6	4 30 finish
10 1 00	1.70	28	3.4			
11 2	1.9.	28	3.6			
12 3	2.0.	28.5	3.9		37	31½
13 4	2.1.	28.5	3.3			
14 5	2.2.	28	2.6			
15 6	2.20	28	2.2	3 lbs. acid		
16 7 10	2.20	27	2.5	separating		
17			2			

Gallons in Fermenter

Stocked with 375 Lbs. of No. 117K Seed.

Yield of Yeast, Net _____ lbs. _____ % Yield

Fermenting strength of yeast _____

Process of Fermentation (A) (C)

30 TIME Hours	% BALLG. 1.00	°C 28.5	ACIDITY 1.5	7.7	25		Feed
1 4	0.90	28	1.8				
2 5	0.85	28	2.5				4 oz (1)
3 6	0.85	28	3.0				
4 7	1.00	28	2.8				6 oz (2)
5 8	1.1.	28	2.7		6		
6 9	1.2.5	28	2.8				10 oz (3)
7 10	1.4.	28	3.0				
8 11	1.5.	28	3.2				2 30 (4)
9 12	1.6.	28	3.3		6		
10 1	1.70	28	3.4				4 30 finish
11 2	1.9.	28	3.6				
12 3	2.0.	28.5	3.9		37		31½
13 4	2.1.4	28.5	3.3				
14 5	2.2.	28	2.6				
15 6	2.120	28	2.2	3 lb. acid			
16 7 10	2 20	27	2.5 2	separating			
17							
18							
19							
20							

Time run to separators, From 7 10 M. to 8 10 M. No. of separators 5

Name _____

Wood Fermenter No. I

NATIONAL GRAIN YEAST CORPORATION

Manufacturing Process—Table

Mash No. M-180 Date 4/27/31

RAW MATERIALS

Malt _____ lbs.
Sprouts _____ lbs.
Corn _____ lbs.
_____ lbs.
Molasses _____ lbs.
_____ lbs.
Am. Phosphate _____ lbs.
Ammonia _____ lbs.
Am. Sulphate _____ lbs.
_____ lbs.

Acid Added

For Neutralization _____
During Fermentation _____
In Liquid Yeast _____

Total Materials _____ lbs.

	Molasses			Grain Mash		
Total gallons molasses and water				Start Filtering		
Sterilizing Temperature				Finish Filtering		
Sterilizing Time	From	M. to	M.	From	M. to	M.
Time start running in fermenter						
Gravity of first extract						
Acidity of first extract						
Gallons in Fermenter						

Stocked with 375 Lbs. of No. 117K Seed

Yield of Yeast, Net _____ lbs. _____ % Yield

Fermenting strength of yeast _____

Process of Fermentation (A) (C)

400 TIME Hours	% BALLG.	°C	ACIDITY	737	25	Feed
1 ✓00	1.20 1.15	28 28	1.5 2.4			
2 6	1.10	28	3.9			5 oz (1)
3 7	1.05	28	3.8			
4 8	1.1	28	4.2			7 oz (2)
5 9	1.2	28	4.0		6 ✓	
6 10	1.3	28	3.6			11 oz (3)
7 11	1.4	28	3.3			
8 12	1.5	28	3.5			3 30 (4)
9 1	1.60	28	3.6		6 ✓	5 30
10 2	1.8	28	3.9			finish
11 3	1.0	30	4.1		✓	

Gallons in Fermenter

Stocked with 375 Lbs. of No. 117K Seed.

Yield of Yeast, Net lbs. % Yield

Fermenting strength of yeast

Process of Fermentation (A) (C)

TIME Hours	% BALLG.	°C	ACIDITY	737	25	Feed
400	1.20	28	1.5			
1 ✓00	1.15	28	2.4	✓	✓	
2. 6	1.10	28	3.3			5[30] (1)
3 7	1.05	28	3.8			
4 8	1.1	28	4.2			7[30] (2)
5 9	1.2	28	4.0		6 ✓	
6 10	1.3	28	3.6			11[30] (3)
7 11	1.4	28	3.3			
8 12	1.5	28	3.5			3[30] (4)
9 1	1.60	28	3.6		6 ✓	
10 2	1.8	28	3.9			5[30]
11 3	1.90	28	3 4			finish ✓
12 4	2.10	28	2.3	2 lts acid.	37	31½ hrs
13 5	2.20	28	2.5			
14 6	2.20	28	2.8	2 lt. acid.		
15 7	2.20	27.5	2.8			
16 8[10]						
17						
18						
19						
20						

Time run to seperators, From 8[10] M. to 9[10] M. No. of seperators 5

Name

Wood Fermenter No. 2

Mash

Wednesday ~~Dec.~~ 7th for
Friday ~~Dec.~~ 9th

1- 1st gen. seed	6- conc Y's
199- 16 (M)	3539 to 3544 Inc

Xtra Mol.

Malt	3,600# ✓	1,500	(1) ✓	Two Molasses Mashes	
Sprouts	3,900	2,400	(2) ✓	each ~~2~~ ~~"~~ Molasses	22½" + 5"
Molasses	23,000			~~2~~ Liters Acid	18 L ✓ + 4 L.
				~~48~~ Ca. Phosphate	48 # ✓ + 6 #

2,500 Gallons (S.C.T.)	16,500 Gallons (Wood Tank)
16 # (C)	257 # (C)
30 # (C)	60 L (B)

	B
1:00 A.M.	19 - cc 12 ✓
2:30 A.M.	19 - cc 14 ✓
4:00 A.M.	20 - cc 16 ✓
5:30 A.M.	17 - cc 18 ✓
7:00 A.M.	20 - cc 22 ✓

	B
100 P.M.	20 I. ✓
3:30 PM - 20.0.	20 " ✓
6:30 PM 20 cc.	14 " ✓
9:30 PM = 20 cc.	12 " ✓
1:00 A.M. 20 - cc	10 ✓
6:00 A.M. 20 - cc	

10:00 A.M. 20 I. ✓

Temp to 170 F.

PLAINTIFF'S EXHIBIT P-15.
Photostat of Defendant's Mash Sheet of December 7 to 9, 1932.

Friday

1- 1st Gen Seed.
199- 1G (M)

6- Conc Yis
3539 to 3544 Inc

Extra Mol.

Two Molasses Mashes

Malt	3,600# ✓	1,500 (1) ✓	each [illegible]" Molasses 22½" + 5"
Sprouts	3,900	2,400 (2) ✓	[illegible] Waters Acid 18 L + 4 L.
Molasses	23,000		[illegible] Ca. Phosphate 48# + 6#.

2,500 Gallons (S.C.T.)

16# (A)
30# (C)

16,500 Gallons (Wood Tank)

257# (C)
60 L (B)

	B		B
1:00 A.M.	19 – cc 12	100 F.M.	20 I.
2:00 A.M.	19 – cc 14	3.30 PM – 20.0	20 "
4:00 A.M.	20 – cc 16	6.30 PM 20.0 cc	16 "
5:30 A.M.	19 – cc 18	9.30 PM = 20.0 cc	12 "
7:00 A.M	20 – cc 22	1:00 A.M. 20 – cc	10
		6:00 A.M. 20 – cc	

10:00 A.M. 20 I.

Heat to 170 F.

PLAINTIFF'S EXHIBIT P-15.
Photostat of Defendant's Mash Sheet of December 7 to 9, 1932.

PROCESS TABLE

Mash No. 3539 Date 12/9/32

RAW MATERIALS

Malt ..... lbs.
Sprouts ..... lbs.
..... lbs.
Molasses ..... lbs.
..... lbs.
A ..... lbs.
B ..... lbs.
C ..... lbs.
D ..... lbs.
C. P. ..... lbs.

Total Materials ..... lbs.

WORT STORAGE

16,500 Gallons Wood.
A ..... lbs.
B ..... lbs. 602 ✓
C 257 lbs. ✓

Gravity 11.9 — % Ball.
Acidity 4.0 — C.C.

B. in Mash ..... lbs.

Stocked with 7/4" J-Y Lbs. of Seed No. 197-26 (5)
Yield—Net Lbs. % Yield
Baking Test—
Remarks—

TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
12 30	1.30	28	1.6		50 ✓		20 ✓		
1								20 ✓	1.30 (3) ✓
2	1.20	28	2.6					15 ✓	
3	1.30	26	2.4	4.54					3 30 (4) ✓
4	1.40	28	2.4	4.54			9 ✓	15 ✓	
5	1.55	28	2.7						5 30 (5) ✓
6	1.75	29	2.1				9 ✓	8 ✓	
7	1.70	20	2.7				9 ✓		7 45 Stop
7 45	1.70	29	2.7						
8									
9									

AT 6 15 ✓ M. STOP COOLERS AND ADD WATER
AT 7 45 M. BACK ½ INTO [redacted] FERMENTER NO. II

10	8 30	29	2.7				10 ✓		8 30 (2) ✓

Baking Test—

Remarks—

	TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
1	12 30	1.30	28	1.6		50 ✓		20 ✓		
2	2	1.20	28	2.6					20 ✓	1.30 (3) ✓
3	3	1.30	28	2.4	4.54				15 ✓	
4	4	1.40	28	2.4	4.54			9 ✓	15 ✓	3 30 (4) ✓
5	5	1.55	28	2.7						5 30 (5) ✓
6	6	1.75	29	2.1				9 ✓	8 ✓	
7	7	1.70	20	2.7				9 ✓		7 45 Stop
8	7 45	1.70	29	2.7						
9										
10	8 30		29	2.7				10 ✓		8 00 (2) ✓
11	9	1.55	29	2.7					8 ✓	
12	10	1.40	28	2.5						8 15 (4) ✓
13	11	1.50	28	2.5				6 ✓		11 15 (3) ✓
14	12	1.65	28	2.5						
15	1 15	1.75	27.5	2.5						11 45 Stop ✓
16										
17										
18										

AT 6 15 ✓ M. STOP COOLERS AND ADD WATER

AT 7 45 ✓ M. BACK ½ INTO [redacted] FERMENTER NO. II

CUT DOWN AIR AT— 11 45 ✓ M. ACID IN WASH WATER 2 ✓ LITERS

TIME FOR SEPARATING—FROM 1 15 ✓ M. TO 1 51 ✓ M.

PUT WASHED YEAST INTO RECEIVER NO. 4

word 4

FERMENTER NO.

PROCESS TABLE

Mash No. 3540 Date 12/9/32

RAW MATERIALS

Malt ..... lbs.
Sprouts ..... lbs.
..... lbs.
Molasses ..... lbs.
..... lbs.
A ..... lbs.
B ..... lbs.
C ..... lbs.
D ..... lbs.
C. P. ..... lbs.

Total Materials ..... lbs.

WORT STORAGE

..... Gallons .....
A ..... lbs. .....
B ..... lbs. .....
C ..... lbs. .....
.....
Gravity ..... % Ballg.
Acidity ..... C.C.

B. in Mash ..... lbs.

Stocked with ~~34~~ "Pure Bud (Y-1) Lbs. of Seed No. 197-26 (2-3)
Yield—Net Lbs. % Yield
Baking Test—
Remarks—

	TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
	1 15	1.30	28	1.6		50 ✓		20 ✓		
1	2 15	1.20	28	2.6					20 ✓	
2	3	1.30	28	2.6	4.24				15 ✓	2 15 (3) ✓
3	4	1.40	28	2.4	4.54					4 15 (4) ✓
4	5	1.50	28	2.4					9 ✓ 15 ✓	
5	6	1.65	28	2.5					9 ✓ 8 ✓	6 15 (5) ✓
6	7	1.80	29	2.7					9 ✓	
7	8	1.75	29	2.1						8 30 ✓ Stop
8	8 30	1.75	29	2.1						
9										

AT 7 ✓ M. STOP COOLERS AND ADD WATER
AT 8 30 M. BACK ½ INTO Lam FERMENTER NO. II

10										
11	10	1.60	29	2.5					10 ✓	8 45 ✓ (2)

Yield—Net ..... Lbs. ..... % Yield

Baking Test—

Remarks—

	TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
	1 15	1.30	28	1.6		50 ✓		20 ✓		
1	2 15	1.20	28	2.6					20 ✓	
2	3	1.30	28	2.6	4.24				15 ✓	2 15 (3) ✓
3	4	1.40	28	2.4	4.54			9 ✓	15 ✓	4 15 (4) ✓
4	5	1.50	28	2.4						
5	6	1.65	28	2.5				9 ✓	8 ✓	6 15 (5) ✓
6	7	1.80	29	2.7				9 ✓		
7	8	1.75	29	2.1						8 30 ✓
8	8 30	1.75	27	2.1						Acty
9										

AT ..... 7 ✓ ..... M. STOP COOLERS AND ADD WATER

AT ..... 8 30 ..... M. BACK ½ INTO ..... FERMENTER NO. II

10										
11	10	1.60	29	2.5				10 ✓		8 45 (2) ✓
12	11	1.50	28	2.4					8 ✓	
13	12	1.65	28	2.4						9 00 (4) ✓
14	1	1.75	28	2.4					6 ✓	
15	1 55	1.85	27	2.4						12 00 (3) ✓
16										12 30 ✓
17										Acty
18										

CUT DOWN AIR AT— 12 30 ✓ M. ACID IN WASH WATER ..... LITERS

TIME FOR SEPARATING—FROM 1 55 ✓ M. TO 2 40 ✓ M.

PUT WASHED YEAST INTO RECEIVER NO. 4

Wood

FERMENTER NO. I

67

PROCESS TABLE

Mash No. 3541 Date 12/9/32

RAW MATERIALS

Malt ..... lbs.
Sprouts ..... lbs.
..... lbs.
Molasses ..... lbs.
..... lbs.
A ..... lbs.
B ..... lbs.
C ..... lbs.
D ..... lbs.
C. P. ..... lbs.

Total Materials ..... lbs.

WORT STORAGE

..... Gallons .....
A ..... lbs. .....
B ..... lbs. .....
C ..... lbs. .....
.....
Gravity ..... % Ballg.
Acidity ..... C.C. .....

B. in Mash ..... lbs.

Stocked with ..... Lbs. of Seed No. .....
Yield—Net ..... Lbs. ..... % Yield
Baking Test— .....
Remarks— .....

	TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
1										
2										
3										
4										
5										
6										
7										
8										
9										

AT ..... M. STOP COOLERS AND ADD WATER

AT ..... M. BACK ½ INTO ..... FERMENTER NO. .....

10										
11	9	1.30	81	2.3						
12	10	1.11	82	1.5						

Stocked with _____ Lbs. of Seed No. _____

Yield—Net _____ Lbs. _____ % Yield

Baking Test—

Remarks—

	TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
1										
2										
3										
4										
5										
6										
7										
8										
9										

AT.....M. STOP COOLERS AND ADD WATER

AT.....M. BACK ½ INTO.....FERMENTER NO.....

10							
11	9	1.30	21	2.3		8	(5)
12	10	1.45	21	2.5	6 15	8 30	(6)
13	11	1.60	28	2.8			
14	12	1.70	28	2.8	6 12	9	(7)
15	1	1.85	28	2.6	9	12	(6)
16	2	1.90	27.5	2.5		12 30	(4)
17	2 40	1.95	27	2.5		1 10	Stop
18							

CUT DOWN AIR AT— 1 10 M. ACID IN WASH WATER _____ LITERS

TIME FOR SEPARATING—FROM 2 40 M. TO 3 55 M.

PUT WASHED YEAST INTO RECEIVER NO. 4

Sun II

FERMENTER NO.....

PROCESS TABLE

Mash No. 3542 Date 12/9/32

RAW MATERIALS

Malt ..... lbs.
Sprouts ..... lbs.
..... lbs.
Molasses ..... lbs.
..... lbs.
A ..... lbs.
B ..... lbs.
C ..... lbs.
D ..... lbs.
C. P. ..... lbs.

Total Materials ..... lbs.

WORT STORAGE

..... Gallons .....
A ..... lbs. .....
B ..... lbs. .....
C ..... lbs. .....
.....
Gravity ..... % Ballg.
Acidity ..... C.C. .....

B. in Mash ..... lbs.

Stocked with 150 # Buck J — 5" J-2 Lbs. of Seed No. 197-2 C (2-1) 197-2 B (J)
Yield—Net ..... Lbs. ..... % Yield
Baking Test— .....
Remarks— .....

	TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
	3 45	1.30	27	1.6		50		20		
1	5	1.10	28	2.6					20	4 45 (2)
2	6	1.20	28	2.05					15	
3	7	1.35	28	2.05					9 15	5 45 (3)
4	8	1.50	28	2.5					9 8	8 45 (4)
5	9	1.65	28	2.7					9	
6	10	1.80	29	2.9					9	11 45 Stop
7	11	1.75	29	3.0						
8	11 45	1.70	29	3.0						
9										

AT 10 45 M. STOP COOLERS AND ADD WATER
AT 11 45 M. BACK ⅓ INTO Open FERMENTER NO. I

10										
11	1	1.45	28	2.6					1	12 = (2)

Stocked with 5" J-2 Lbs. of Seed No. 197-2E (1-1)

Yield—Net ..... Lbs. 197-2E (2-1) % Yield

Baking Test—

Remarks—

	TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
	3 45	1.30	27	1.6		50		20		
1	5	1.10	28	2.6					20	4 45 (2)
2	6	1.20	28	2.6					15	
3	7	1.35	28	2.65				9	15	5 45 (3)
4	8	1.50	28	2.5				9	8	8 45 (4)
5	9	1.65	28	2.7				9		11 45 Stop
6	10	1.80	29	2.9						
7	11	1.75	29	3.0						
8	11 45	1.70	29	3.0						
9										

AT 10 45 M. STOP COOLERS AND ADD WATER

AT 11 45 M. BACK ½ INTO Ferm FERMENTER NO. 1

10										
11	1	1.45	28	2.6				6		12 45 (2)
12	2	1.45	28	2.6				3		
13	3	1.55	28.5	2.6				4	5	1 45 (4)
14	4	1.65	28.5	2.6				1	9	3 45 (3)
15	5	1.80	28.5	2.7						4 45 (2)
16	6 30	1.90	27	2.6						5 45 Stop
17										
18										

CUT DOWN AIR AT— 5 00 M. ACID IN WASH WATER 9 LITERS

TIME FOR SEPARATING—FROM 6 30 M. TO 7 00 M.

PUT WASHED YEAST INTO RECEIVER NO. 3

Wood 2

FERMENTER NO.

PROCESS TABLE

Mash No. 3543 Date 12/9/32

RAW MATERIALS

Malt ..... lbs.
Sprouts ..... lbs.
..... lbs.
Molasses ..... lbs.
..... lbs.
A ..... lbs.
B ..... lbs.
C ..... lbs.
D ..... lbs.
C. P. ..... lbs.

Total Materials ..... lbs.

WORT STORAGE

..... Gallons .....
A ..... lbs. .....
B ..... lbs. .....
C ..... lbs. .....
.....
Gravity ..... % Ballg.
Acidity ..... C.C. .....

B. in Mash ..... lbs.

Stocked with 6¼" ✓ - 2 Lbs. of Seed No. 197-2G(r)

Yield—Net ..... Lbs. ..... % Yield

Baking Test— .....

Remarks— .....

	TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
	4 30	1.13	27	1.6		50 ✓		20 ✓		
1	6	1.10	28	2.6				20 ✓		5 30 ② ✓
2	7	1.25	28	2.3				15 ✓		
3	8	1.40	28	2.3						6 30 ③ ✓
4	9	1.55	28	2.5				9 ✓ 15 ✓		
5	10	1.65	28	2.9						9 30 ④ ✓
6	11	1.75	29	3.0				9 ✓ 8 ✓		
7	12	1.75	29	3.0						12 30 ✓ stop
8	12 32	1.70	29	3.0				9 ✓		
9								✓		

AT 11 00 ✓ M. STOP COOLERS AND ADD WATER

AT 12 30 ✓ M. BACK ½ INTO Iron FERMENTER NO. I

10										
11	2	1.45	28	2.5				✓		12 45 ⑤

Stocked with 6¼" V-2 Lbs. of Seed No. 197-2G(V)

Yield—Net Lbs. % Yield

Baking Test—

Remarks—

	TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
	4 30	1.3	27	1.6		50		20		
1										
2	6	1.10	28	2.6					20	5 30 (2)
3	7	1.25	28	2.3					15	
4	8	1.40	28	2.3						6 30 (3)
5	9	1.55	28	2.5					9 15	
6	10	1.65	28	2.9						9 30 (4)
7	11	1.75	29	3.0					9 8	
8	12	1.75	29	3.0						12 30 Stop
9	12 32	1.70	29	3.0					9	

AT 11 00 M. STOP COOLERS AND ADD WATER

AT 12 30 M. BACK ½ INTO Iron FERMENTER NO. I

10										
11	2	1.45	28	2.5					6	12 45 (2)
12	3	1.45	28	2.5					4 45	1 45 (4)
13	4	1.55	28	2.5					9	2 45 (3)
14	5	1.65	28.5	2.6						4 45 (2)
15	6	1.75	28	2.6						5 45 Stop
16	7 10	1.85	27	2.6						
17										
18										

CUT DOWN AIR AT— 5 45 M. ACID IN WASH WATER LITERS

TIME FOR SEPARATING—FROM 7 10 M. TO 7 50 M.

PUT WASHED YEAST INTO RECEIVER NO. 3

Wood 3

FERMENTER NO.

PROCESS TABLE

Mash No. 3544 Date 12/9/32

RAW MATERIALS

Malt ..... lbs.
Sprouts ..... lbs.
..... lbs.
Molasses ..... lbs.
..... lbs.
A ..... lbs.
B ..... lbs.
C ..... lbs.
D ..... lbs.
C. P. ..... lbs.

Total Materials ..... lbs.

WORT STORAGE

..... Gallons.....
A ..... lbs. .....
B ..... lbs. .....
C ..... lbs. .....
.....
Gravity ..... % Ballg.
Acidity ..... C.C.....

B. in Mash ..... lbs.

Stocked with ..... Lbs. of Seed No. .....
Yield—Net ..... Lbs. ..... % Yield
Baking Test— .....
Remarks— .....
.....

TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
1									
2									
3									
4									
5									
6									
7									
8									
9									

AT ..... M. STOP COOLERS AND ADD WATER

AT ..... M. BACK ½ INTO ..... FERMENTER NO. .....

10

Yield—Net ..... Lbs. ..... % Yield

Baking Test— .....

Remarks— .....

.....

	TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
1										
2										
3										
4										
5										
6										
7										
8										
9										

AT..... M. STOP COOLERS AND ADD WATER

AT..... M. BACK ½ INTO..... FERMENTER NO.....

10										
11	1	1.30	28	2.4					10 ✓	11 50 (3) ✓
12	2	1.45	28.5	2.6					10 ✓	1 20 (6) ✓
13	3	1.55	28.5	2.6					10 ✓	2 50 (6) ✓
14	4	1.70	28	2.6					6 ✓	4 20 (6) ✓
15	5	1.80	28	2.6						5 20 (4) ✓
16	6	1.90	28	2.6						6 20 Stop ✓
17	7	1.95	28	2.5						
18	7 50	1.95	27	2.5						

CUT DOWN AIR AT— 6 20 ✓ M. ACID IN WASH WATER ..... LITERS

TIME FOR SEPARATING—FROM 7 50 M. TO ..... M. .....

PUT WASHED YEAST INTO RECEIVER NO. 3 .....

.....

FERMENTER NO. I

Seed

PROCESS TABLE

Mash No. 199-1G (M)

Date 12/9/32

RAW MATERIALS

Malt ..... lbs.
Sprouts ..... lbs.
..... lbs.
Molasses ..... lbs.
..... lbs.
A ..... lbs.
B ..... lbs.
C ..... lbs.
D ..... lbs.
C. P. ..... lbs.

Total Materials ..... lbs.

WORT STORAGE

2500 Gallons S.C.T
A 16 lbs. ✓
B ..... lbs.
C 30 lbs. ✓
.....
Gravity 13.0 - % Ballg.
Acidity 8.0 - C.C.

B. in Mash 60 lbs.

Stocked with Copper Prof. P.C. Lbs. of Seed No. 3300 gals
Yield—Net Lbs. % Yield
Baking Test—
Remarks— Press Report
M-3533-34-35-36- 11 presses 1" M-3537-38-4-7 presses 7"

	TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
	11 00	6.20	27	3.6		25 ✓		85 ✓		
1	12	5.90	27	4.0						
2	1	5.70	27	4.7						
3	2	5.30	27	5.6						
4	3	5.00	27	6.4				2 Pails ✓		
5	4	4.68	27	7.3				3 " ✓	15 ✓	
6	5	4.10	27	8.0				4 " ✓		
7	6	3.60	27	9.5				4 " ✓	15 ✓	
8	7	3.40	27	9.5				4 " ✓		
9	8	3.20	27	9.0				4 " ✓	15 ✓	
								160 lbs in Dkg		
	AT ..... M. STOP COOLERS AND ADD WATER									
	AT ..... M. BACK ½ INTO ..... FERMENTER NO.									
10										

Seed-199-1G
82 :: J-1

12 30 (2) ✓
1 30 (3) ✓
2 30 (4) ✓
4 30 (5) ✓
5 30 (6) ✓
8 00 (3) ✓
9 00 Stop ✓

Stocked with Compressed Yeast I.C. Lbs. of Seed No. 3300 gals

Yield—Net Lbs. % Yield

Baking Test—

Remarks— Press Report

M-35 33-34-35-36- 11 presses 1" M-35 37-38-4-7 presses 7"

Seed-199-18
32"-J-1

	TIME Hours	GRAVITY % BALLG.	°C	ACIDITY	pH	A	B	C	D	ACID ADDED
	11 00	6.20	27	3.6		25 ✓		85 ✓		
1	12	5.90	27	4.0						
2	1	5.70	27	4.7						
3	2	5.30	27	5.6						
4	3	5.00	27	6.4						
5	4	4.60	27	7.3						
6	5	4.10	27	8.0						
7	6	3.60	27	7.5						
8	7	3.40	27	9.5						
9	8	3.20	27	9.0						
10										
11	9	3.20	27	9.0						
12	10	3.20	27	8.0						
13	10 30	3.20	27	8.0						
14										
15										
16										
17										
18										

2 Pail ✓
3 " 15 ✓
4 "
4 " 15 ✓
4 "
4 " 15 ✓
4 "
60 lbs in Pail

12 30 (2) ✓
1 30 (3) ✓
2 30 (4) ✓
4 30 (5) ✓
5 30 (6) ✓
8 00 (3) ✓
9 00 Stop ✓

AT..... M. STOP COOLERS AND ADD WATER

AT..... M. BACK ½ INTO..... FERMENTER NO.....

1st Gen Seed - Circulate + Wash in
Iron 2 " I + Copper tank No. I
Steam out lines at 9 30 PM

CUT DOWN AIR AT— 9 00 M. ✓ ACID IN WASH WATER 2 ✓ LITERS

TIME FOR SEPARATING—FROM 10 30 ✓ M. TO 11 30 ✓ M.

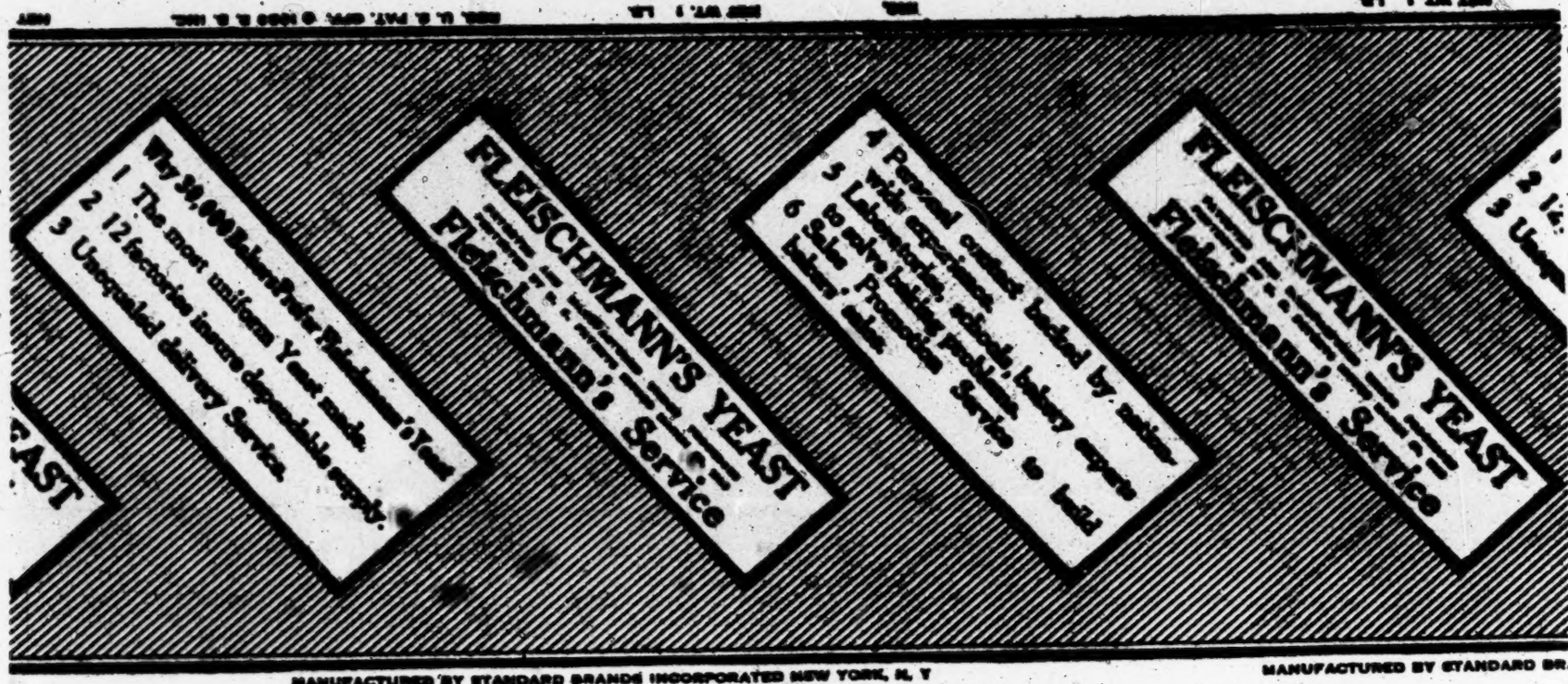
PUT WASHED YEAST INTO RECEIVER NO.

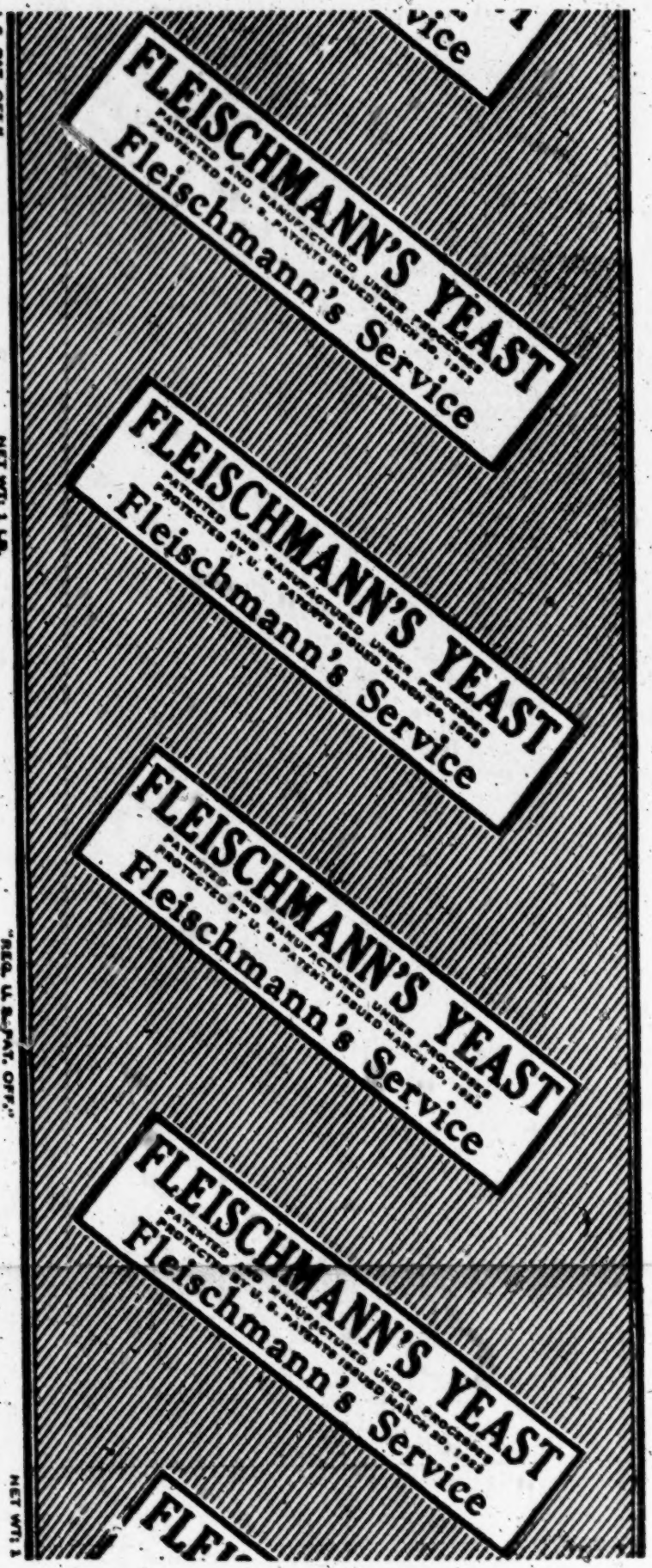
Iron

FERMENTER NO. I

PLAINTIFF'S EXHIBIT P-16.

Wrapper Labeled "Fleischmann Yeast, Patented and Manufactured Under Processes Protected by United States Patents Issued March 20, 1923."




FLEISCHMANN'S YEAST
PATENTED AND MANUFACTURED UNDER PROCESSES PROTECTED BY U. S. PATENTS ISSUED MARCH 20, 1923
Fleischmann's Service
NET WT. 1 LB.
"REG. U. S. PAT. OFF."

Form 42-11-A Jan-11-21

LIBERTY YEAST CORPORATION

(_____ PLANT

YEAST MANUFACTURE PROCESS-TABLES

Mash No. 784

Date May 19 1923

Raw Materials		
		lbs.
		lbs.
Beet molasses	Gals.	lbs.
For neutralization	Liters.	lbs.
Ammonium Phosphate	30	lbs.
Aqua Ammonia	66	lbs.
Total Material		lbs.
Filtercel	43	lbs.

Total Gallons Molasses and Water	
Sterilizing Temperature	
Sterilizing Time. From . m. to . m.	
Time start running in fermenter	
Gravity of first extract	20.4
Acidity " " "	27 c c
Gallons in fermenter	100 -114 56.33
Amount of Stock: lbs. and brand	100 -115

Acid added to liquid yeast obtained _____ liters.

Time of pressing yeast _____ hours.

Amount of yeast obtained, net _____ lbs. _____ % yield

Fermenting strength of yeast

Process of Fermentation.

TIME Half	Hours	% BALLG.	TEMPERATURE	ACIDITY	ADDITIONS H_2SO_4	ADDITIONS NH_4OH	
	6 PM	18	24	19	10 c	3 c	12
1	7 30	18	25	23			12
2	8 30	17	26	27			12
3	9 30	16	27	32		3 c	12
4	10 30	15	27	36		3 c	12
5	11 30	14	27	40		3 c	12
6	12 00	13	27	44	4 c	3 c	12
7	1 30	13	27	46	2	3 —	—
8	2 00	13	27	37	2 c	3 — 2 c	12
9	3 00	14	27	20	4 c	2 —	12
10	4 30	15	27	15	5 c	2 c	12

Mash I is

Photostat Ed P

Amount of Stock: lbs. and brand 100 - 115

Acid added to liquid yeast obtained _____ liters.

Time of pressing yeast _____ hours.

Amount of yeast obtained, net _____ lbs. _____ % yield

Fermenting strength of yeast

Process of Fermentation.

	TIME Hrs	Hours	BALLG.	TEMPERATURE	ACIDITY	ADDITIONS H_2SO_4	ADDITIONS NH_4OH	
	6	PM	18	24	19	10 c	3 c	12
1	7	30	18	25	23			12
2	8	30	17	26	27			12
3	9	30	16	27	32		3 c	12
4	10	30	15	27	36		3 c	12
5	11	30	14	27	40		3 c	12
6	12	00	13	27	44	4 c	3 c	12
7	1	30	13	27	46	2	3 c	—
8	2	00	13	27	37	2 c	3 c 2 c	12
9	3	00	14	27	20	4 c	3 c 2 c	12
10	4	30	15	27	15	5 c	2 c	12
11	6	00	17	27	24	5 c	1 c	12
12	6	30	17	27	29	5 c	2 c	12
13	7	30	21	27	34	2 c		12
14	8	30	23	27	42	5 c		12
15	9	30	23	27	30			12
16	10	30	23	27	27			12
17	11	30	23	27	25			12
18	12	00 PM	23	23	20			
19								
20								
21								

Mash In

Run

Roll

Time run to separators 12 30 P m.

PLAINTIFF'S EXHIBIT P-17.
Photostat Entitled Liberty Yeast Corporation, Dated May 19, 1923.

Form 43-2m-4-22

LIBERTY YEAST CORPORATION

(GLOBE PLANT)

YEAST MANUFACTURE PROCESS-TABLES

Seed Yeast No.

Mash No. 1333 Raw Materials Date Jan. 8th 1923.

Corn	709	lbs.			
Malt	776	lbs.			
Sprouts	440	lbs.	H_2SO_4		
Rye	8	lbs.	For Neutralization	1333	cc
Beet molasses Gals.	2334	lbs.	During Fermentation	6000	cc
		lbs.	Added to Liquid Yeast	1300	cc
Ammonium Phosphate	50	lbs.			
Aqua Ammonia	20	lbs.			
	20	lbs.			
Total Material		lbs.	153 @ 4:15 A.M.		

	BEET MOLASSES	GRAIN MASH
Total Gallons Molasses and Water		Pumped to filter M. 7:50 A.M.
Sterilizing Temperature		Finished filtering M. 14.5
Sterilizing Time From M. to M.		Last water °Blg 25.5
Time start running in fermenter		10944
Gravity of first extract		# 421
Acidity " " "		
Gallons in fermenter		
Stocked with	240 #	

Amount of yeast obtained, net lbs. 6449-50 46.5 % yield

Fermenting strength of yeast 26.6 1330-31-32-33-34

Process of Fermentation. January 9th, 1923.

TIME Half	Hours	BALLG.	TEMPERATURE	ACIDITY	ADDITIONS H_2SO_4	ADDITIONS NH_4OH
	10:00 A.M.	3.6	20	4.7		
1	11:00	3.5	20.5	3.4		
2	12:00	3.5	21.5	3.2		
3	1:00	3.3	22.5	3.5		
4	2:00	3.1	23.5	3.9		
5	3:00	2.8	25.	4.7	Shift Change	
6	4:00	2.4	26.	5.9		5
7	5:00	1.9	27	7.0		
8	6:00	1.7	28	7.5		
9	7:00	1.6	28	6.8		

Photosta

Sterilizing time: From M. to M.		
Time start running in fermenter		7:50 A.M.
Gravity of first extract		14.3
Acidity " " "		25.5
Gallons in fermenter		10944
Stocked with	240 #	# 421

Amount of yeast obtained, net lbs. 46.5 % yield

Fermenting strength of yeast 26.6 6449-50 1330-31-32-33-34

Process of Fermentation. January 9th, 1923.

	TIME Hall	Hours	BALLG.	TEMPERATURE	ACIDITY	ADDITIONS H 2 SO 4	ADDITIONS NH 4 OH
	10:00	A.M.	3.6	20	4.7		
1	11:00		3.5	20.5	3.4		
2	12:00		3.5	21.5	3.2		
3	1:00		3.3	22.5	3.5		
4	2:00		3.1	23.5	3.9		
						Shift Change	
5	3:00		2.8	25.	4.7		
6	4:00		2.4	26	5.9		5
7	5:00		1.9	27	7.0		
8	6:00		1.7	28	7.5		
9	7:00		1.6	28	6.8		
10	8:00		1.6	28	6.4		5
11	9:00		1.6	28	5.6		
12	10:00		1.6	28.5	4.2		
						Shift Change	
13	11:00		1.6	28.5	2.8	2	
14	12:00		1.6	28	2.8	2	
15	1:00		1.6	28	2.8	2	
16	2:00		1.6	28	3.0		
17							
18							

Time run to separators, From 2:00 A m. to 3:20 A m. No. of separators 13.

Name

Fermenter No. 12.

PLAINTIFF'S EXHIBIT P-18.

Photostat of Liberty Yeast Corporation "Mash Sheet", (Globe Plant) Dated January 8, 1923.

PLAINTIFF'S EXHIBIT P-19.
Dr. Lamb's Calculation.

Claydick 105

I. A wort of 12° Balling is prepared (say 2500 gallons).

II. A part (650 gallons) of this wort is diluted in the fermenter with water to 1° Balling. That is, water is added until the volume is 12 times as great i.e. 7800 gals. These selected volumes obey the rule as to mean balling — thus

7800 × 1 = 7800
1850 × 12 = 22200
9650
30,000
9650 | 30000 (3,1

to the diluted wort (II).

III. Seed yeast is added, aeration started, and the feed of concentrated wort (12° - 1850 gallons left) into the dilute solution is started and maintained. The final volume will be 9650 gallons.

IV. yeast is harvested.

PLAINTIFF'S EXHIBIT P-20.

Sheet Describing Experiment Which Dr. Schultz Carried Out to Replace Portion of Molasses Mash by Alcohol, and Etc.

Fermentation Laboratory

Dr. Frey

— Molasses check and molasses plus alcohol run for yields of molasses in the presence of alcohol.

33% Cane
67% Beet molasses
+.75% Ammonium acid phosphate
+.9% Sodium bicarbonate

Hrs.	% Molasses 17 at start	300 gms. molasses	225 gms. molasses + 29 gms. C_2H_5OH equiv. to 38 gms. 95%
1	6	pH	pH
2	8	about	about
3	10	4.5	4.5
4	12		
5	14		
6	15		
7	13		
7½	5		
8	—		
Total NH_3 equi.		28.5	31.0
Protein on Solids		50.7	53.2
Yield 26% Solids		104.3	104.4
Bake		92, 66, 76	91, 61, 63

The above experiment where the zulauf finishes one-half hour from the end of the growing period shows that the same yield was obtained from 29 gms. C_2H_5OH as 75 grams molasses.

A. Schultz

PLAINTIFF'S EXHIBIT P-21.

Sheet Explaining Tests by Dr. Lamb.

NEUTRALIZING POWER OF AMMONIUM LACTATE.

1. Prepared a wort, corresponding to that in the propagating vat of M-177 at the end of the 6th hour, without souring and without yeast.

2. To one portion of this wort I added the amount of lactic acid corresponding to the initial acidities. The calculated amount of phosphoric acid which would be inherent from the ammonium phosphate was then added. Finally the amount of sulphuric acid corresponding to the added ammonium sulphate was added. The concentration of the hydrogen ion was measured by means of the quinhydron electrode after each of the above additions.

3. This experiment was then repeated with an initial addition of ammonium lactate corresponding to the amount of ammonium lactate that was added in the vat of M-177 at the end of the 6th hour.
At the end of the 6th hour we have 1575# materials in the fermenter in 9850 gallons. This calculates to 6.1 grams in 318 c.c.
At this place also we have 3.3 pounds of lactic acid equivalent to 2.8 c.c. of .05 N; 85# ammonium acid phosphate equivalent to 2.7 c.c. 10% phosphoric acid and finally 47.7# ammonium sulphate equivalent to 13.6 c.c. 0.2 N $H_2S\ O_4$.
The ammonium lactate in the wort was 2%. This was made by adding theoretical quantities of ammonia and lactic acid.

The table below gives the corresponding PH's.

	Without Ammonium Lactate	With Amm. Lac.
6.1 grs. mash materials in 318 c.c.	4.92	5.07
+ 2.8 c.c. .05 N lactic	4.81	4.94
+ 2.7 c.c. Phosphoric (10%)	3.50	3.70
2 c.c. H_2SO_4 (.2N)	3.34	3.55
4 c.c.	3.21	3.47
6 c.c.	3.12	3.36
8 c.c.	3.00	3.25
10 c.c.	2.90	3.16
12 c.c.	2.80	3.05
13.6 c.c.	2.73	3.00
16.0 c.c.		2.87
18.0 c.c.		2.80
20.0 c.c.		2.70

PLAINTIFF'S EXHIBIT P-22.
Table Showing Prior Art Disclosure.

	cited in Patent Office or by defendant in former litigation	amount of inorganic nitrogen from ammonium salts which might release harmful acid during yeast manufacture	any stated or remedied acidity problem from inorganic ammonium salts during yeast manufacture	any substantial use of neutralizing aqua ammonia as source of assimilable nitrogen during yeast manufacture	Any disclosure like defendant's process (Plaintiff's Exhibits 13, 14, 15) in respect of (a) substantial use of inorganic salts as assimilable nitrogen source in yeast manufacture	Any disclosure like defendant's process (Plaintiff's Exhibits 13, 14, 15) in respect of (b) consequent acidity problem, and use of neutralizing aqua ammonia or other anti-acid in yeast manufacture	(c) correlated dilute starting solution and concentrated reserve solution, with regulated feed of latter at rate proportional to yeast propagation without any significant alcohol formation not consumed by yeast before propagation ended	yield of yeast on raw materials on same scale used by plaintiff and defendant
LAVEDAN	too late in date	unstated- low yield shows no significant amounts	unstated- low yield shows no acidity problem	no	no	no	no	about 33% on molasses
POLSKY	yes	negligible	no, because stated chemical composition of salts made no difference	no	no	no	no	asserts 90%
JACQUEMIN	yes	unstated, except says may be omitted without difference in result, therefore insignificant	no	no	no	no	no	seed yeast only, and yield unstated
MOLHANT	yes	unstated - no suggestion of any significant amount	no	no	no	no	no	no yield stated seed yeast and merely acclimatized to acid for making alcohol
ROTH	yes	no suggestion of any significant amount and says equivalent with none	no	no	no	no	no	preparatory treatment only
POLLAK	yes	none	no	yes	no	no	no	preparatory treatment only
FRITSCHE	yes	suggests adding only 1/3 - 1/1x amount of salts used by defendant	no, low yield shows no acidity problem	no	yes	no	no	unstated, except increase of 7 to 12% yeast with much alcohol
HENNEBERG Jour. Soc. Chem. Ind. Pure Products	yes	unstated, and experimental	no	no	unstated, and experimental	not clearly stated even in distilled water; clearly stated none in ordinary water commercially used	no	unstated
Brennar Centralblatt	no	unstated, and experimental	No, says acid harmful to molds, and yeast practice so used acid to prevent infection	no	no, experimental on molds	no	no	molds, but yields not stated
WILFERT PRESSHEFE	no	none	no	no	no	no	no	preparatory

PLAINTIFF'S EXHIBIT P-23.

British Patent No. 778 of 1880 to William Robert Lake.

[Second Edition.]

A.D. 1880, 21*st* FEBRUARY. N° 778.

Manufacture of Yeast.

LETTERS PATENT to William Robert Lake, of the Firm of Haseltine, Lake, and Co., Patent Agents, Southampton Buildings, London, for an Invention of "IMPROVEMENTS IN THE MANUFACTURE OF YEAST." A communication from abroad by Jacques Rainer, of Vienna, Austria, Doctor of Chemistry.

PROVISIONAL SPECIFICATION left by the said William Robert Lake at the Office of the Commissioners of Patents on the 21st February 1880.

WILLIAM ROBERT LAKE, of the Firm of Haseltine, Lake, and Co., Patent Agents, Southampton Buildings, London. "IMPROVEMENTS IN THE MANUFACTURE OF YEAST." [A communication from abroad by Jacques Rainer, of Vienna, Austria, Doctor of Chemistry.]

This Invention relates to the manufacture of yeast without the ordinary alcoholic fermentation and without the ordinary formation of secondary products.

The said improvements consist,—

First. In the employment of vegetable peptones with the addition of small quantities of dextrose, maltose, or other similar hydro-carbons for the purpose of the culture and growth of the cells of yeast.

Secondly. In adding successively peptones and sugar to the comparatively stable nutritious solution used for growing the yeast, and in successively withdrawing the finished yeast produced in the said solution.

Thirdly. In the manufacture of pressed yeast in large quantities without alcoholic fermentation and without production of alcohol or vinegar.

Fourthly. In the admission of ozonized air or ozonized oxygen for the purpose of accelerating and intensifying the growth of the yeast.

According to the present improvements the process of manufacturing the yeast is carried into practice in the following manner, that is to say:—

The vegetable albuminous matters obtained from maize or other grain legumen, or from waste products, such as bran, the radicles of malt, spent malt, gluten, and other similar products are treated with water containing a small quantity of an alkali or of a mineral acid, and the extract is peptonised by the addition of diastase in the presence of diluted acid. In the radicles of malt especially part of the albuminous matters are already peptonised by the previous growing of the barley,

and their extract already contains some diastase. According to the quantity of peptones contained in the fluid a quantity of sugar, dextrose, maltose, or of their equivalents is added in the proportion of about one-half of the weight of dry peptones present. This addition ought to be about one half per cent. of the total quantity of the fluid, and serves solely for the purpose of yielding the material for the formation of the structure of the cells of yeast. A small quantity of beer yeast or pressed yeast is also added to start the growth, and atmospheric air or ozone is conducted into the fluid to accelerate the growth and impede fermentation. When after some hours the sugar already added has been consumed by the growth of the yeast, a further portion of about from one-tenth to one-fourth per cent. is again added, and in a similar manner the peptones already consumed are replaced by gradual and successive addition of further small quantities of peptones.

The yeast produced may be likewise successively and gradually removed by skimming it off or by allowing it to settle.

The purification and pressing of the yeast takes place in the usual manner.

SPECIFICATION in pursuance of the conditions of the Letters Patent filed by the said William Robert Lake in the Great Seal Patent Office on the 21st August 1880.

WILLIAM ROBERT LAKE, of the Firm of Haseltine, Lake, & Co., Patent Agents, Southampton Buildings, London. "IMPROVEMENTS IN THE MANUFACTURE OF YEAST." A communication from abroad by Jacques Rainer, of Vienna, Austria, Doctor of Chemistry.

This Invention relates to the manufacture of yeast without the ordinary alcoholic fermentation, and without the ordinary formation of subsidiary products.

The said improvements consist,—

Firstly. In the employment of vegetable peptones with the addition of slight quantities of dextrose, maltose, or other similar hydrocarbons for the purpose of the culture and growth of the cells of yeast.

Secondly. In adding successively peptones and sugar to the comparatively stable nutritious solution used for growing the yeast, and successively withdrawing the finished yeast produced in the same.

Thirdly, In a process or method of manufacturing compressed yeast in large quantities without alcoholic fermentation, and without any subsidiary production of alcohol or vinegar.

Fourthly. In the admission of ozonised air or ozonised oxygen for the purpose of acclerating and intensifying the growth of the yeast.

According to the present improvements the process of manufacturing the yeast may be carried into practice in the following manner, that is to say :—

The vegetable albuminous matters obtained from maize or other grain or legumen, or from waste products, such as bran, the radicles of malt, spent malt, gluten, and other similar products are treated with water containing a small quantity of an alkali or of a mineral acid, and the extract is peptonised by the addition of diastase in the presence of diluted acid. In the radicles of malt especially part of the albuminous matters is already peptonized, and their extract already contains some diastase. According to the quantity of peptone contained in the fluid sugar, dextrose, maltose, or their equivalent is added in the proportion of about one half of the weight of dry peptones present. This addition ought to be about one half per cent. of the total quantity of the fluid, and serves solely for yielding the material for the formation of the structure of the cells of yeast. A small quantity of beer-yeast or pressed yeast is also added to start the growth, and atmospheric air or ozone is conducted into the fluid to accelerate such growth. When after some hours the sugar already added has been consumed by the growth of the yeast, a further quantity of about one tenth to one fourth per cent. is again added, and in a similar manner the peptones already consumed are replaced by the gradual and successive addition of further small quantities.

The yeast produced may be successively and gradually removed by skimming or by allowing it to settle. The purification and pressing of the yeast take place in the usual manner.

The process of manufacturing the compressed yeast commercially is as follows :—

The vegetable albuminous substances in the corn cereals or other vegetables, or such refuse of industrial establishments as bran cornings, malt residuum, gluten and the like are extracted with the aid of from 15 to 20 parts by measure of water, made slightly alkaline. They are then either peptonised by adding an excess of lactic acid (about 4 per cent.) or mineral acids (about $\frac{1}{4}$ per cent. of phosphoric acid, or about $\frac{4}{10}$ per cent. of either sulphuric acid or hydrochloric acid) at a temperature of from 85 to 100 degrees Fahrenheit, or they are at once macerated in dilute solutions of the above acids and simultaneously converted into peptone.

Lake's Improvements in the Manufacture of Yeast.

A portion of the albuminous substances (from 5 to 10 per cent. of the total weight) in the dried cornings will be already transformed into peptone by the process of vegetation. The albuminous substances in cereals, maize, or other vegetables, and in bran and malt residuum are transformed into peptone by the addition of diastase. In order to effect the conversion it is sufficient to add to one part by weight of the albuminous matter when dry one part by weight of dry malt, or five parts by weight of cornings. As stated the liquid in which the albuminous matter is to be transformed into peptone must contain lactic acid (4 per cent.), phosphoric acid (as much as $^1/_4$ per cent.), sulphuric acid or hydrochloric acid (about $^3/_4$ per cent.), because the presence of an acid is absolutely necessary in the process of converting these substances into peptone.

A temperature of about 100 degrees Fahrenheit is the most suitable for the conversion of said substances into peptone, and a period of from 18 to 20 hours will be sufficient to effect it. It may however be also carried out at lower temperatures during a correspondingly longer time. In working cornings it is superfluous to add malt, because the diastase contained in the cornings is more than sufficient for the process of conversion into peptone. Therefore it is only necessary in this case to use one of the above named acids in the proportions given.

The mixture of the cornings with water may be effected by hand or by means of a stirring apparatus actuated by any suitable machinery. In mashing malt or cereals, that is to say, in preparing the necessary maltose, dextrose, or the like, I use the apparatus ordinarily employed for such process, care being taken to maintain the temperature and other conditions necessary for proper concentration, as described and recommended in modern technical journals especially in Professor Dr. Max Maerker's "Handbuch der Spiritus Fabrication" (Handbook of the Manufacture of Spirits).

The slimy pectates contained in the cornings as well as in other materials are dissolved by the combination of diastase and acids.

When the preparation of pure peptone is acquired the pectates may be separated by an endosmotic apparatus or dialysator in such a manner that the peptone is dialysed through proper membranes in water, while the gelatinous pectates remain as a residuum.

The acids are neutralized by means of soda, or by saturating the liquid with basic phosphate of lime in the manner hereinafter described.

The prepared peptone liquid, with or without a percentage of sugar, may be shipped as a saleable article, or it may be delivered in a dry state, or as a syrup or extract obtained by boiling the liquid down in a water bath, by steam, or preferably in a vacuum.

The liquid containing peptone may be separated from solid matter (hydrocarbons vegetable fibre, or the like) by simple extraction, maceration, or pressure, or by centrifugal action, or it may be carefully cleaned by filtration or settling. It is advisable however before cleaning by filtering or settling to neutralize any acid present by means of soda, or to saturate the liquid with basic phosphate of lime, the latter being preferable because the phosphoric acid required by the yeast is thus abundantly furnished to it.

In order to start the growth of the yeast gelatinized starch is added after being transformed in the usual way into dextrose by boiling with an addition of mineral acids. In the place of starch thus prepared an addition may be made of maltose, molasses, or sugar mixed with beer yeast or compressed yeast. The amount thus added should correspond to the percentage of peptone in the liquid, being one half of the dry weight of the peptone. The said hydro carbons should however always be only from $^1/_2$ to 1 per cent. of the weight of the entire liquid, and should even then serve exclusively for the formation of the walls of the cells of the yeast.

The vegetation of the yeast will take place most satisfactorily at temperatures varying from 57 to 64 degrees Fahrenheit. At a higher temperature losses may easily occur by reason of the partial conversion of the sugar used into coagulated lactic acid or into alcoholic fermentation, instead of furnishing the yeast with

substance for cells. The yeast is either propagated, as is the custom in Holland, in shallow vessels in which the depth of liquid is about five inches, so that a sufficient quantity of atmospheric air has access thereto; or it may be better and more safely effected in vats made of wood, glass, masonry, cement, or other suitable material, into which atmospheric air is conducted by suitable distributors through tubes or pipes by means of blowers or compressors in a fine state of division.

Instead of atmospheric air alone it is more advantageous to use air containing an increased amount of ozone or of oxygen partially converted into ozone. The latter is prepared by successively adding hydrogen dioxide to the propagating liquid. The percentage of ozone in the air is increased by means of phosphorus, or by causing it to pass through a closed vessel in which permanganate of potassa is mixed with the necessary quantity of mineral acid. The air thus enriched with ozone is thus allowed to pass into the propagating liquid.

The growth of the yeast will be completed within from 6 to 8 hours after every sufficient addition of dextrose, maltose, or other material, according to the density of the propagating liquid used, the temperature of the latter, and the amount of the ozone in the air. The percentage of peptone of the mass may amount to from 1 to 2 per cent. or more of its weight, while only from half to one per cent. of dextrose or other hydro carbons is added at each time, in order to be sure to prevent the formation of coagulated lactic acid or alcoholic fermentation, and the losses attendant upon it for my process.

When the entire amount or bulk of the dextrose or other sugar added to promote the growth of the yeast has been consumed after from 6 to 8 hours, a further quantity thereof, say, from $^1/_{20}$ to $^1/_{10}$ per cent. is added. The peptone may also, after having been consumed, be added in portions, or may be allowed to flow in gradually and continuously. The same propagating liquid made by successive replacement of the matter consumed remains in use for weeks or months, unless it is rendered impure by other substances, or by subsiding fermentation, and thus made unfit for further use.

In the same manner as the materials necessary for the propagation of the yeast are added the yeast produced may be successively withdrawn, and only the yeast suspended in the liquid remains behind as the germ for the ferments of alcohol to be afterwards formed.

The yeast is obtained either by skimming it from the surface of the liquid or by separating it from the propagating liquid by filtration, or finally by gathering it after tapping the vats from the bottom upon which it is deposited in a compact layer.

In working on a large scale it is advisable to place the vats in terraced batteries in order to effect the transfer of the propagating liquid from one vessel to the other with facility.

In order to produce yeast as free as possible from subsidiary ferments the propagating liquid may be prepared in a more dilute state, that is to say, with a percentage of peptone of only from $^3/_4$ to 1 per cent. The hydrocarbons (dextrose, maltose, or the like) may also be added in smaller quantities for example, as a first dose about $^1/_2$ per cent., and then every 3 hours about $^1/_{20}$ per cent.

The greater part of the peptone present will then be transformed into yeast in from 12 to 15 hours, a sufficient supply of pure air, if necessary conducted through sulphuric acid or oxygen containing ozone, being provided, and the entire process being carried on at a temperature varying from 54 to 63 degrees Fahrenheit. The whole liquid is then cooled by a suitable apparatus, or by adding cold water or ice; the best temperature being from 45 to 50 degrees Fahrenheit. Within from 36 to 48 hours the yeast obtained will settle on the bottom of the vat. The propagating liquid may be allowed to flow away. The yeast obtained by this improved process is purified and condensed in the usual manner, but in order to increase its durability phosphate of lime amounting to from 4 to 5 per cent. of the total weight of the yeast to be made may be added before compressing it.

Experience has shown that from 250 to 300 parts of pure and active compressed

yeast may be obtained from 100 parts of pure peptone. For the growth of the said quantity of yeast only about 200 parts of dextrose or sugar are required.

Having thus fully described the said Invention as communicated to me by my foreign correspondent and the manner of performing the same, I wish it understood that I claim,—

First. The process of cultivating the cells of yeast from vegetable peptone by the addition of dextrose or similar hydrocarbons, substantially as specified.

Second. In making yeast, the process comprising the successive additions of peptone and hydro-carbons to the propagating liquid, together with the successive removals of the yeast produced, all substantially in the manner and in the proportions specified.

Third. The process or method herein described of manufacturing compressed yeast without alcoholic fermentation, and without producing alcohol or vinegar, as above set forth.

Fourth. In making yeast the application of currents of ozone to the propagating liquid for the purpose of accelerating the growth of the yeast, and of increasing the intensity of its development, substantially as specified.

In witness whereof, I, the said William Robert Lake, have hereunto set my hand and seal, this Twenty-first day of August, in the year of our Lord One thousand eight hundred and eighty.

WM. ROBT. LAKE. (L.S.)

Redhill: Printed for His Majesty's Stationery Office, by Love & Malcomson, Ltd.
[G. 9781—25—3/1907.]

PLAINTIFF'S EXHIBIT P-24.

[Translation by George C. O. Haas, Ph.D., New York]

Delbruck "Illustriertes Brennerei-Lexikon"

(Pages 347-349)

Article "Hefefabrikation"

Yeast-manufacture is the production of bakers' yeast on a factory scale in yeast factories. In the present state of the art it is generally carried out, in its practical execution, according to two processes, which in turn manifest differences within themselves in method of operation. In the case of both processes a mash that is rich in yeast nutrients is first produced from raw materials, especially grain, that contain starch or sugar and are rich in proteins.* In the case of one of the two processes, the so-called old process (also called the Vienna or skimming process) the yeast is then cultivated directly in this mash containing grains and is extracted from it, after the development has been completed, by decanting or skimming off from the surface. The alcohol that is produced in the course of the manufacturing process is of good quality and is worked up into beverage spirits. As by-product in the distilling of the mash there results a valuable hot residual liquid. On the basis of the observations and experiences gained in the old process with regard to the production of commercial yeast that is capable of being shipped, a new method of production of compressed yeast, which is called air yeast-manufacture or wort yeast-manufacture, has developed in the course of the last 25 years from the scientific discovery that the multiplication of yeast is exceedingly stimulated by introduction of air and that by means of the blowing-in

* [Translator's note.—The word *zuckerhaliigen* is a misprint for *zuckerhaltigen*, and *eiweissweichen* for *eiweissreichen*.]

of air into a wort pitched with yeast the yield of yeast from the same quantity of raw material, in comparison with the former yield, can be increased to more than three times as much. In this process the mash is not pitched directly with yeast, but a wort is extracted from it by means of clarification or by filtering in filter presses, and this wort is set in fermentation with yeast, in which connection large quantities of air are supplied to the fermenting yeast during the entire time of its development by blowing it into the wort. After the development has been completed, the yeast is extracted from the wort by allowing it to settle on flat settling pans or by centrifugalizing with the use of separators. The wort that has been freed from yeast is subjected to distillation, in which process the alcohol is extracted from it. In consequence of the abundant introduction of air into the fermenting wort, the alcohol is greatly contaminated by aldehyde and other admixtures, so that in most cases it can only be employed for technical purposes and be utilized in a less valuable manner than the alcohol from the old process. The yield of alcohol in the aeration process is also materially less than in the latter. Because of its great dilution and its low content of digestible nutritive substances the distilled wort has only a slight feed value. It is therefore employed, if at all, only as a pouring-over liquid at the mixing of the feed or passes into the waste liquors of the plant. A valuable agricultural by-product in connection with the wort process is constituted by the grains that remain behind at the clarifying or filtering of the mash. Computed on the initial material, they possess about half the value of the residual liquid in the old process.

The advantages of the aeration process in comparison with the old process lie primarily in the unequally greater yield of yeast, the lower cost of production, the

simpler mode of procedure, and the greater uniformity and certainty of operation. The manufacture, which has in recent years been extended to large-scale operation in almost all air-yeast factories, turns out in Germany at the present time about 95 per cent of the entire production of yeast. The old process is still adhered to in the smaller and medium-sized yeast distilleries, primarily agricultural ones, in consideration of the superior quality of spirituous liquors and the advantages of the residual liquid, which is rich in nutritious substances, as an excellent hot feed for fattening and milk-production.

I. In the "old process" the principal raw materials are: kiln-dried malt, rye, barley, maize, clarified residual liquid, and water, sometimes also potatoes and green malt. The grain is first subjected to a good dry cleaning and is then thoroughly comminuted in mills, roller mills, or roller crushers. In the preparation of the mash, in which process 125-150 kilograms of total grain are ordinarily used for 1000 liters of mash and the malt constitutes about 25-30 per cent, the principles that generally obtain for the dissolving, peptonizing, making up into paste, and saccharification are followed (see Mash). As mashing temperatures there are employed the temperatures between 45 and 62° C. which are the most favorable for the peptonizing and saccharification, in which connection those raw materials which require a higher mashing temperature in accordance with the nature of their starch (maize, dried potatoes, barley) are previously hydrolyzed and made into a paste by boiling or else by the application of slight pressure (1 to 1½ atmospheres). After a saccharification time of 2 to 3 hours at 60-62° C. the mash is cooled off. In order to promote the dissolving and peptonizing of the proteins, it receives, during the cooling, an addition of several liters of mash that is acid

with lactic acid or of a fairly large part of the clarified residual liquid. Between 50 and 54° C. the cooling is discontinued, and the mash remains standing at this temperature for about 1 to 2 hours. Thereupon there is effected a rapid cooling down to the pitching temperature, which is to be chosen, according to the circumstances, between 21 and 24° C. For pitching there serves the yeast that has been cultivated in a special mash (cf. Yeast Mash) or compressed yeast, sometimes also both of these (cf. Pitching and Pitching Yeast). The mash is mixed in the fermenting vat with the rest of the clarified residual liquid and receives in connection therewith an addition of sulfuric acid. After its completion the mash has a sugar content of 8 to 12° Balling and an acidity of between 0.3 and 0.4 (in working with residual liquid, 0.5 to 0.6). During the fermentation the yeast rises upward in the mash (rising tendency) with the aid of the carbonic acid developed and gradually collects upon the surface of the mash in a blanket of foam about 30 centimeters high. In 12 to 13 hours the yeast foam has attained its greatest height, and it now remains stationary, since the development of carbonic acid subsides. After the initially lighter bubbles of a glassy appearance have thereupon within 2 to 3 hours become milkily cloudy and less readily movable, and the yeast cells larger and more uniform, the yeast is ready for the skimming-off. The attenuation of the mash has at this stage progressed as far as 3 to 4° Balling. In the case of normal vats the skimming-off of the yeast is concluded after 5 to 6 hours. The yeast that is removed is at once mixed with cold water and thereupon freed by straining (cf. Yeast Strainer) from the grain and mash particles still adhering to it and then passed into flat washing and settling vats. After the water has been changed two to three times, the thickly pasty yeast that has settled

down is freed from the still adherent water by pressing in sacks or filter presses (cf. Yeast Press) and brought to the consistency requisite for shipment. The yeast that has been pressed dry has a dry substance of 25 to 27 per cent. For sale at retail the yeast is shaped into measured pound pieces (cf. Yeast Form and Dividing Machine); for wholesale shipment it is customary to pack it in tightly rammed and pressed bags (see Yeast Bag Press).

The yields attained in the skimming process are widely different in the individual factories according to the installation, management of the plant, and mode of operation. On an average 12 to 13 kilograms of yeast and 14 to 15 liters of alcohol are obtained from 100 kilograms of grain that are doughed in.

II. In the air-yeast process there are used as raw materials especially malt (mostly green malt), maize, and malt sprouts. Sometimes potatoes, rye, raw barley, molasses, and sugar-beet juice also constitute the principal component of the mash material. In addition to the content of starch and sugar, a high protein content of the raw materials is particularly necessary. The percentage composition thereof is subject to not inconsiderable fluctuations according to the price of raw materials, the purpose of the manufacturing process, and the installation available for the treatment in the individual plants. On an average one chooses 40 to 60 per cent of maize, 30 to 40 per cent of barley in the form of green malt, and 10 to 25 per cent of malt sprouts, in which connection the maize may also be replaced by other materials containing starch, such as potatoes, raw barley, or manioc. For the preparation of the mash the raw materials are previously comminuted in crushers or wet mills and mixed in the fore-mashing vat with slightly acidified water of 40 to 45° C., and the mash liquid thereupon heated with the

steamed or boiled portion of the raw materials (maize, potatoes, raw grain) with vigorous mashing and with gradual increase of the temperature as far as 50° C. After standing for 1 to 2 hours there follows the scalding with steam to the saccharification temperature of 62 to 63° C., at which the mash now remains standing for about 2 to 3 hours for saccharification. The clarifying material (malt sprouts, oat husks, chaff) may be added before the mashing or after the saccharification. After the saccharification is completed, the mash, which as a rule has a concentration of 10 to 12° Balling, is subjected to a peptonization for 12 to 15 hours, with the employment of pure lactic acid bacteria, and is thereupon passed through filter presses or clarified for extraction of the wort. The solid constituents of the mash (grains) are extracted with hot water (afterflushing) until all the soluble extract substances are transferred into the wort. The latter, after adequate cooling in the fermenting vat, is set for fermentation with about 4 to 6 per cent of pressed-dry yeast (computed on the basis of 100 kilograms of raw material) at favorable growth temperatures, 22-25° C., and at the same time vigorously aerated during the entire time of the fermentation, in order to promote the growth of the yeast. The fermentation and development of the yeast are completed in 8 to 10 hours. Toward the end of the fermentation the quantity of air is diminished. The yeast is extracted from the fermented wort by separating (cf. Separator) or by allowing it to settle upon flat settling pans, and is pressed dry in filter presses to the consistency requisite for shipment. The alcohol is extracted by distillation from the wort that has been freed from yeast. Under normal circumstances 28 to 30 kilograms of yeast and 18 to 20 liters of alcohol are obtained from 100 kilograms of doughing-in material. By means of great dilution of the

wort, increased aeration at a lower temperature, and increase of the proteins the yeast yield may be further augmented, while the alcohol yield then declines. (Lg.)

Original of Pages 347 to 349 of Delbrück "Illustriertes Brennerei-Lexikon."

(Physical Exhibit.)

PLAINTIFF'S EXHIBIT P-25.

United States Patent No. 2,008,584 to Harold L. King, Dated July 16, 1935.

Patented July 16, 1935

2,008,584

UNITED STATES PATENT OFFICE

2,008,584

PROCESS OF TREATING DISTILLERY SLOP

Harold L. King, Bloomfield, N. J., assignor to National Grain Yeast Corporation, Belleville, N. J., a corporation of New Jersey

No Drawing. Application February 4, 1931, Serial No. 513,460

7 Claims. (Cl. 195—29)

My invention is a process of treating waste distillery slop, to render the same available as a constituent of the nutrient medium for the propagation of commercial compressed yeast, and a nutrient medium for the propagation of commercial compressed yeast.

Distillery slop, obtained from the alcoholic fermentation of beet molasses, or blackstrap molasses, has long been regarded as a waste product. This waste product, in the operation of large plants, presents serious difficulties in connection with its disposal. Due to its characteristic odor, it is frequently impossible to discharge the same into the ordinary sewage system of a town or city, and separate sewage systems have been built for its disposal, at a large cost. Attempts have been made to utilize this product in the production of fertilizer, by incinerating the same for obtaining the potassium content, but such method is not economical or profitable. As far as I am aware, no successful profitable method has been devised for utilizing this waste distillery slop.

I have discovered that the waste distillery slop, when properly treated in accordance with my method, may be successfully used as a constituent of a nutrient medium for the propagation of yeast.

When a regular molasses mash of high gravity (22° to 25° Balling) is fermented for the production of commercial alcohol, the yeast present functions chiefly to split up the inverted sugars into ethyl alcohol and carbon dioxide gas. Due to the almost complete exclusion of oxygen, especially after fermentation has set in vigorously, there is only a very slight propagation of yeast cells and, consequently, very little assimulation by the yeast cells, of the large amount of nitrogen matter and dissolved mineral substances present in the mash. This is due to the fact that yeast cannot assimilate the substances necessary for growth and rapid reproduction, except in the presence of large amounts of oxygen. In the absence of oxygen, yeast functions in an entirely different manner, serving as a true agent of fermentation, with a very small degree of cell reproduction.

It therefore, follows that at the end of the fermentation process, and after the fermentable sugars have all been transformed into alcohol and carbonic acid gas, a very large percentage of the original nitrogen matter and mineral salts still remain in the residue or slop, and this slop, if properly treated, is capable of forming a constituent of a nutrient medium for the propagation of yeast.

However, the contents of this original slop and especially the nitrogenous matter is of a more or less complex composition and not easily available for yeast growth, without preliminary treatment to reduce the same to a mass having constituents of a less complex composition.

The slop is to be used in conjunction with a yeast mash formula made up of beet or cane molasses, malt, and malt sprouts.

By actual tests, from a given amount of raw materials, (malt, malt sprouts and molasses) used in conjunction with slop, a quantity of final compressed yeast was obtained which was greater by approximately 20% than the amount which was obtained from the same given amount of raw materials without the use of slop.

The introduction of the slop into the regular mash formula of grain and molasses is accomplished as follows: The total amount of malt to be used is mashed and converted in water, by the usual method, using only a small amount of water, or the minimum amount which can be employed to produce the condition necessary for complete conversion of the malt mash. After conversion is completed, this mash is cooled to a proper temperature of from 128° to 130° F. and a pure culture of lactic acid bacteria is introduced into the mash, the amount of the bacteria to be added, depending to some extent upon the total quantity of raw materials to be soured, as is well known in the art. The mash is held at this temperature of 128° to 130° F., for several hours, (from 3 to 6 hours) until the action of the bacteria has gotten well started and lactic acid is being formed. At this point, the mash is very thick and heavy as only enough water has been added to allow the necessary reactions for complete conversion of the starch and protein of the mash.

The next step in the process is to introduce the waste slop into the souring malt mash, and as dilution proceeds, malt sprouts are gradually added until all of the sprouts have been introduced. As the waste slop is usually added hot, just as it is discharged from the beer still, the mash must be continually cooled to maintain the same at the proper souring temperature of from 128° to 130° F.

About one-half of the total amount of slop to be used in the complete mash, is added to the malt and sprout mash at this point, and the whole mixture is allowed to sour for the balance of the first 24 hours, with the occasional addition of aqua ammonia as the acidification proceeds. The action of the bacteria becomes greatly retarded when the maximum point of acidity is reached, this point, shown by tests, to be that which requires between 40 to 60 c. c. of $\frac{1}{10}$ normal alkali solution to neutralize 40 c. c. of the mash. Therefore, the ammonia is added to keep this acid level considerably below this maximum point, as, for example, between the limits of acidity requiring from 10 to 20 c. c. of the $\frac{1}{10}$ normal alkali to neutralize 40 c. c. of the mash.

In the meantime, in another vat, the beet molasses to be used is treated separately. Instead of using water to dilute the heavy beet molasses, I use the remaining one-half part of the waste slop. The resultant mixture is rendered slightly acid by the addition of sulphuric acid and heated by direct contact with steam to almost a boiling temperature, between 90° to 95° C. Instead of using beet molasses, I may use cane molasses, which would be clarified by the method well known in the distillery art.

The phosphate present in the added slop will produce a precipitation of the albuminous matter. The mash is allowed to set for a suitable length of time, such as over night, so that the precipitates will settle to the bottom of the mixture and the mixture is clarified.

After the precipitated matter has thus settled to the bottom, the mixture is clear. The clear liquid is drawn off, leaving the solids at the bottom.

The clear liquid thus produced is now introduced into the souring grain mash, previously prepared, and fresh culture of lactic acid bacteria is now introduced into the resultant mash, which is allowed to continue to sour for 24 hours more, with the continued addition of aqua ammonia, at intervals, as acidity develops, to keep the acid level between the limits of 10 to 20 c. c. of the $\frac{1}{10}$ normal alkali solution required to neutralize 40 c. c. of the mash. While it is preferred to use aqua ammonia as the alkaline neutralizing agent, yet I may employ other alkaline neutralizing agents, such as lime (CaO) or like substances, with some degree of success. When the souring has proceeded for a sufficient length of time to produce the desired changes in the composition of the mash, ordinarily from 36 to 48 hours, the addition of the alkaline neutralizing agent is discontinued and the amount of acidity required in the completed mash is allowed to accumulate. The final degree of acidity must necessarily depend upon the particular process of yeast cultivation to be employed from this point on. The acidity is determined by the usual method of titration with $\frac{1}{10}$ normal alkali solution on a measured quantity (40 c. c.) of the mash, using specially prepared litmus paper as indicator.

The resultant mash is now sterilized and filtered for separation of the soluble extracted matter from the insoluble grain residues, and the liquid mash is next filter-pressed for more complete clarification by the removal of all finely divided insoluble matter, which is precipitated from the waste slop.

Considering malt, sprouts, and molasses as constituting 100 percent of the regular mash formula, I, preferably, employ 20 parts by weight of malt, 20 parts by weight of sprouts, and 60 parts by weight of molasses, although the proportions may be widely varied. The slop is added in each instance, as a substitute for water, to dilute the mash to a desired gravity. The final mash has a gravity of from 18° to 24° B. This mash contains grain constituents such as the malt and sprouts, these constituents containing easily assimilable nitrogen in the form of amino-acids in an amount sufficient to initiate the lactic acid fermentation, or in other words to supply the lactic acid bacteria during the first part of the process.

During the lactic acid fermentation, described in connection with the grain mash, and also in connection with the combined grain mash and beet molasses mash, the complex protein bodies contained in the slop, as well as those present in the regular mash materials, are split up into nitrogen compounds of much more simple composition and rendered more easily assimilable by the yeast cells, as food. One of the most valuable of the simpler forms of organic nitrogen bodies are the so-called amino-acids. The nitrogen of the amino-acids is easily and readily assimilated by yeast. The composition of the above mash, during the prolonged action of the lactic acid bacteria shows an ever increasing quantity of amino-nitrogen, brought about by the continued breaking down of the more complex proteins. It is, therefore, desirable to prolong the action of the lactic acid bacteria, but the bacteria can only function in the presence of a limited amount of acid, and as the acidity increases, the several reactions incidental to its growth and acid-forming properties are necessarily greatly retarded, and finally completely stopped. In order to obtain the prolonged reactions incidental to the maximum growth of the acid bacteria, the degree of acidity is controlled by the addition of the aqua ammonia, directly to the souring mixture, as explained. Acidity tests are made at intervals and measured amounts of aqua ammonia added. By this procedure, the several chemical and biological changes produced by the acid bacteria are greatly prolonged, resulting in a greatly increased amount of nitrogen matter readily available for rapid assimilation by the growing yeast cells. Particular attention is called to the fact that the aqua ammonia is added to the souring mash, at intervals during the souring period, for the purpose of controlling or regulating the acidity, and thereby prolonging the development or growth of acid bacteria, and all reactions incidental to this growth.

The addition of aqua ammonia to the souring mash is also valuable from another standpoint, as it combines directly with the lactic acid, to produce ammonium lactate, which remains dissolved in the extract of the mash, and, furthermore, increases the amount of nitrogen available for yeast food, during the period of yeast cultivation.

The resultant clear wort, although quite highly colored from the use of the slop, contains only soluble coloring matter which can be readily washed away from the finished yeast and a very good quality of yeast of superior fermenting power, and a very good color obtained. To obtain the best color of the finished product, it is necessary to closely regulate the degree of acidity produced throughout the period of yeast propagation. If the proper concentration of H-ions is maintained during the yeast cultivation period and the same controlled so as to be constant, or nearly so, especially during the latter part of this process, a product of very fine color will result.

The control of the acidity during this very important stage of yeast cultivation is made comparatively simple in connection with the use of the materials as prepared by this process, for the following reason. The addition of aqua ammonia during the prolonged period of souring, and the resultant combination of same with the various organic acids formed, result in an accumulation of chemical substances with the so-called Buffer properties. Namely, these substances restrict and hold within desirable limits the formation of free hydrogen ions or ionizable acid radicals which is very important especially during the latter stages of yeast propagation. In this case, it is possible to maintain the pH concentration at practically a constant level with the addition of small quantities of mineral salts and with the entire elimination of any neutralizing agents during the entire period of yeast cultivation.

While I have described a process in which aqua ammonia is added to the mash containing waste slop, during the souring stage, for regulating the acidity, yet, the invention is not necessarily restricted to the treatment of a mash containing the waste slop, as I contemplate treating yeast mash free from the added waste slop, during the souring period, for controlling or regulating the acidity.

It is to be understood that the form of my invention herewith shown and described, is to be taken as a preferred example of the same, and that various changes in the order of the steps of the method may be resorted to, and equivalents employed, without departing from the spirit of my invention or the scope of the subjoined claims.

Having thus described my invention, what I claim is:

1. The herein described process of treating waste distillery slop to render the same available as a constituent of the nutrient medium for the propagation of commercial compressed yeast, comprising combining the slop with a yeast mash including grain constituents containing easily assimilable nitrogen in an amount sufficient to initiate lactic acid fermentation, adding to the mass thus obtained lactic acid bacteria to effect souring of the same, regulating the degree of acidity of the mass by the addition of aqua ammonia so that the lactic acid bacteria are propagated for a prolonged period and the constituents of the slop thereby rendered available as a nutrient for yeast, sterilizing the resultant mass, and filtering for removing foreign solid matter.

2. The herein described process of treating waste distillery slop to render the same available as a constituent of the nutrient medium for the propagation of commercial compressed yeast, comprising combining the slop with a yeast mash including grain constituents containing easily assimilable nitrogen in an amount sufficient to initiate lactic acid fermentation, adding to the mass thus obtained lactic acid bacteria to effect souring of the same, regulating the degree of acidity of the mass by the addition of an alkaline neutralizing agent so that the lactic acid bacteria are propagated for a prolonged period and the contents of the slop thereby rendered available as a nutrient for yeast, sterilizing the resultant mass, and filtering the same for removing foreign solid matter.

3. In the herein described process of treating waste distillery slop to render the same available as a constituent of the nutrient medium for the propagation of commercial compressed yeast, the steps of combining the slop with a yeast mash including grain constituents containing easily assimilable nitrogen in an amount sufficient to initiate lactic acid fermentation, adding to the mass thus obtained lactic acid bacteria to effect souring of the same, and regulating the degree of acidity of the mass by the addition of an alkaline neutralizing agent so that the lactic acid bacteria are propagated for a prolonged period and the contents of the slop thereby rendered available as a nutrient for yeast.

4. The method of producing a nutrient medium for the propagation of commercial compressed yeast, comprising combining waste distillery slop with a yeast mash including grain constituents containing easily assimilable nitrogen in an amount sufficient to initiate lactic acid fermentation adding to the mass lactic acid bacteria to effect a souring of the same, regulating the degree of acidity of the mass by the addition of an alkaline neutralizing agent so that the lactic acid bacteria are propagated for a prolonged period, sterilizing the mash, and filtering the same.

5. The herein described process of treating waste distillery slop to render the same available as a constituent of the nutrient medium for the propagation of commercial compressed yeast, comprising combining a portion of the slop with a grain mash, adding lactic acid bacteria to the resultant mass to effect a souring of the same, controlling the degree of acidity of the mass by adding thereto an alkaline neutralizing agent so that the lactic acid bacteria are propagated for a prolonged period and the constituents of the slop thereby rendered available as a nutrient for yeast, adding a portion of the slop to a molasses and treating the same for producing a clarified liquid mass, combining the clarified liquid mass with the mass first produced for obtaining a resultant liquid mass, adding additional lactic acid bacteria to the resultant mass for effecting a further souring of the same, regulating the degree of acidity of such resultant mass by the addition of an alkaline neutralizing agent so that the lactic acid bacteria are propagated for a prolonged period and the contents of the slop rendered available as a nutrient for yeast, sterilizing the resultant mass, and filtering.

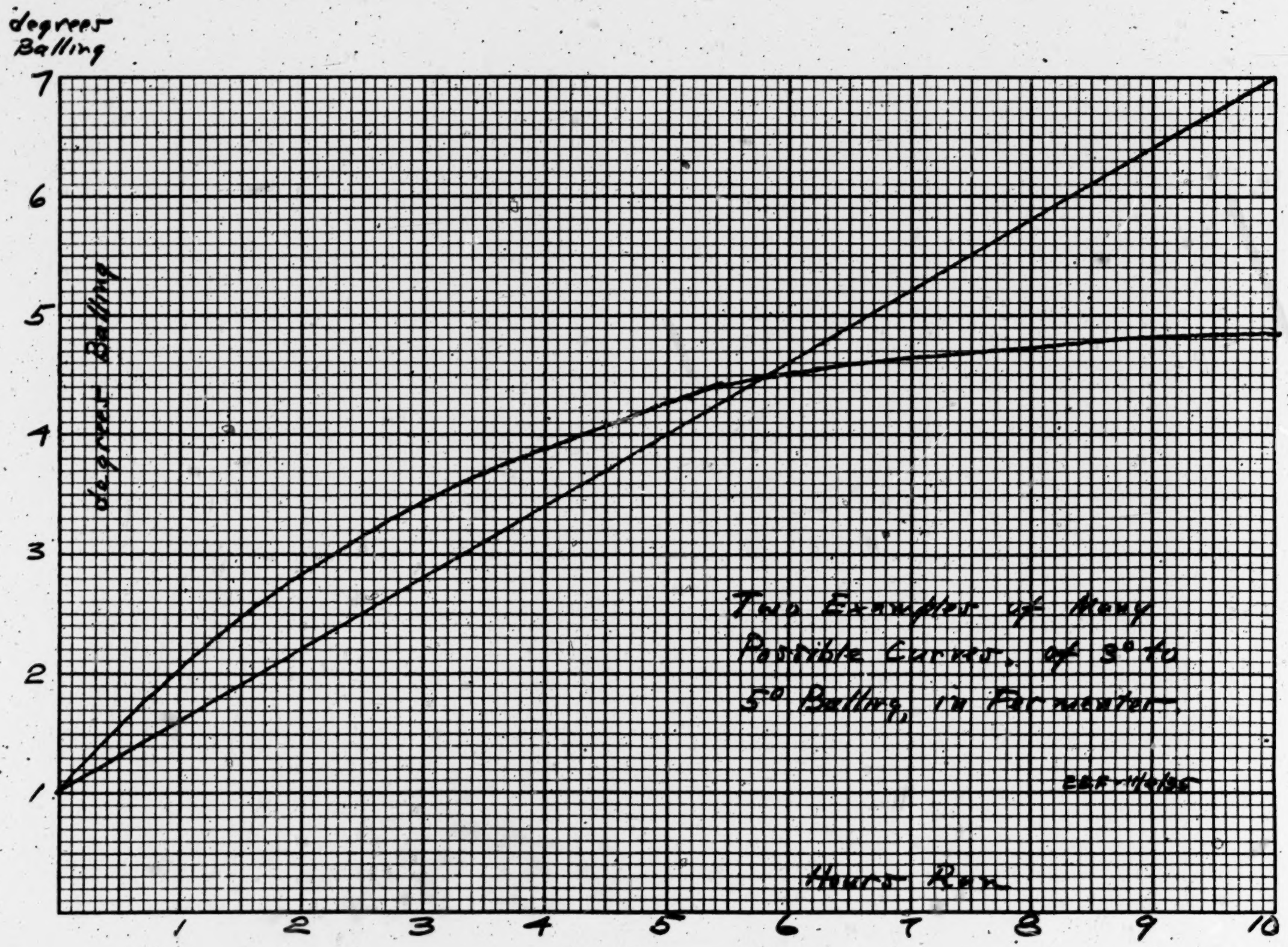
6. The herein described process of treating waste distillery slop to render the same available as a constituent of the nutrient medium for the propagation of commercial compressed yeast, comprising combining a portion of the slop with a grain yeast mash, adding lactic acid bacteria to the resultant mass to effect a souring of the same, controlling the degree of acidity of the mass by adding thereto aqua ammonia so that the lactic acid bacteria are propagated for a prolonged period and the constituents of the slop thereby rendered available as a nutrient for yeast, adding a portion of the slop to beet molasses to dilute the same for providing a second liquid mass, rendering the second liquid mass slightly acid, heating the second liquid mass thus treated and separating the liquid constituent from the solid constituents, combining the liquid constituent of the second mass with the first mass, adding additional lactic acid bacteria to the resultant mass, for effecting a souring of the same, controlling the degree of acidity of the resultant mass by adding thereto aqua ammonia so that the lactic acid bacteria are propagated for a prolonged period and the constituents of the slop thereby rendered available as a nutrient for yeast, sterilizing the resultant mass, and filtering.

7. The herein described process of treating

waste distillery slop to render the same available as a constituent of a nutrient medium for the propagation of commercial compressed yeast, comprising the steps of adding lactic acid bacteria to a grain yeast mash and effecting the propagation of such bacteria within the mash, adding the slop to such mash so that the propagation of the lactic acid bacteria continues and the resultant mass is soured, and regulating the acidity of the mass by the addition thereto of aqua ammonia so that the propagation of the lactic acid bacteria is continued for a prolonged period and the constituents of the slop are rendered available as a nutrient for yeast.

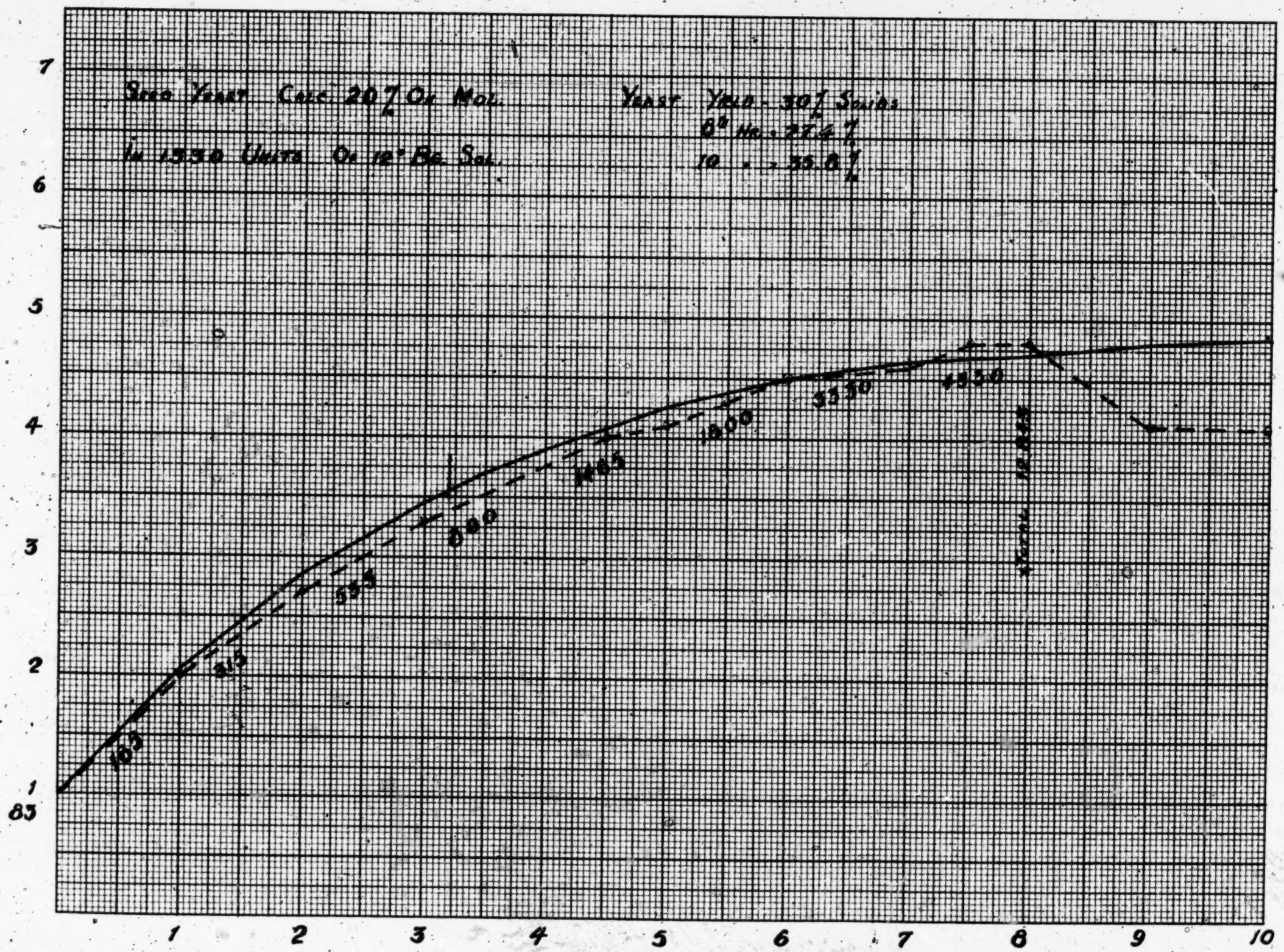
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PLAINTIFF'S EXHIBIT P-26.
Photostat of Free Curves.

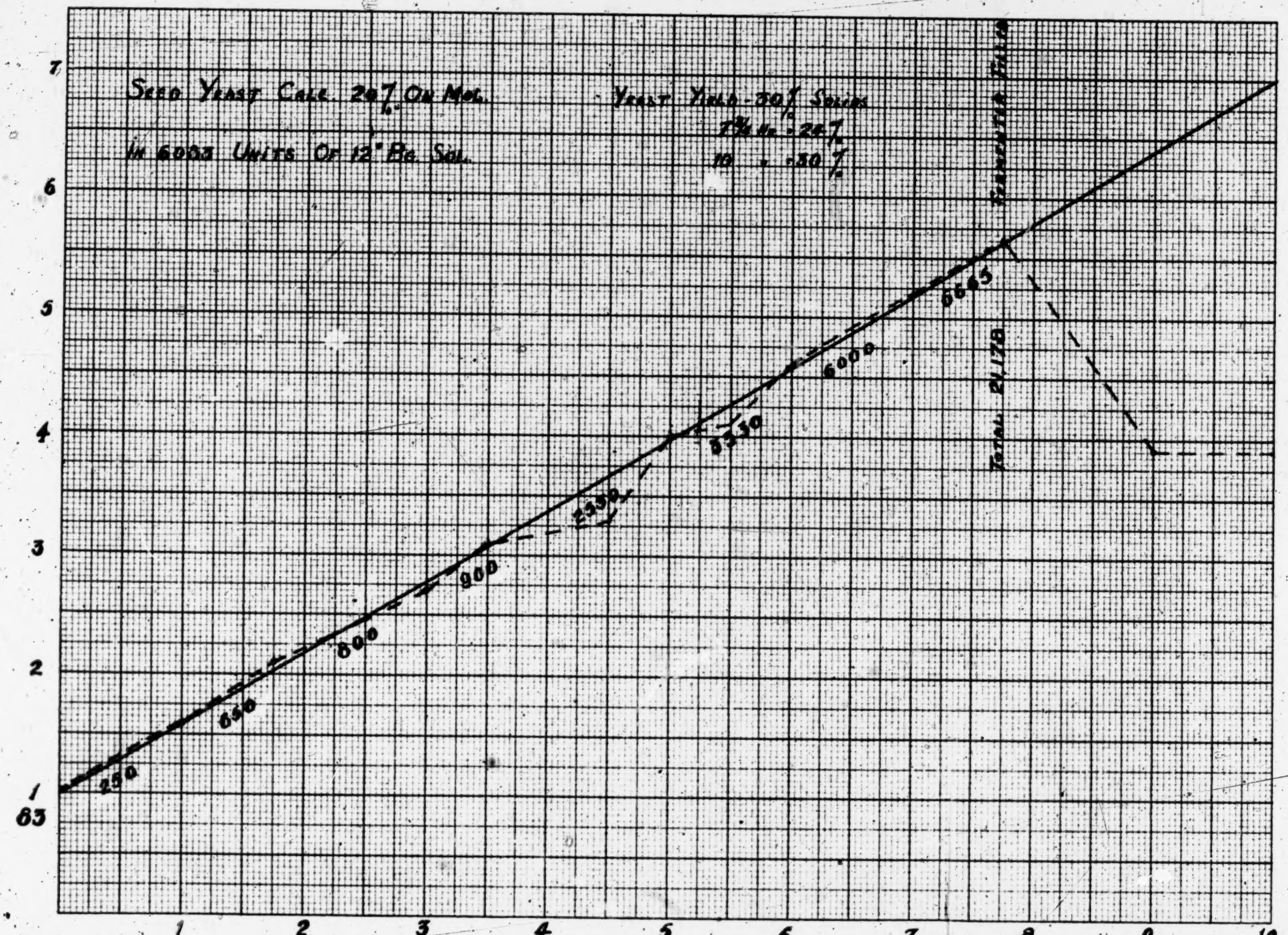


PLAINTIFF'S EXHIBIT P-27.

Graph Giving Arcuate Curve by Dr. Lamb.



PLAINTIFF'S EXHIBIT P-28.
Graph Giving Rectilinear by Dr. Lamb.



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UNITED STATES PATENT OFFICE

LUCIEN LAVEDAN, OF NEW ORLEANS, LOUISIANA.

PROCESS OF MANUFACTURING YEAST.

1,201,062. Specification of Letters Patent. Patented Oct. 10, 1916.

Application filed February 12, 1916. Serial No. 77,896.

To all whom it may concern:

Be it known that I, LUCIEN LAVEDAN, a citizen of the United States, and a resident of New Orleans, in the parish of Orleans and State of Louisiana, have invented certain new and useful Improvements in Processes of Manufacturing Yeast, of which the following is a specification.

My invention relates to improvements in processes of manufacturing yeast, and it consists in the various steps hereinafter enumerated.

The object of my invention is to produce yeast in such a manner that the highest possible yield is effected from a given amount of clear sugared liquid, thereby greatly decreasing the cost of manufacture and making use of certain portions of the raw material which have heretofore been wasted.

A further object of my invention is to provide a process for making yeast which is continuous, and in which there is a continuous aeration of the yeast, thereby supplying to the yeast all the air which is necessary to its maximum development and thus tending to a maximum yield of yeast.

A further object of my invention is to provide a process in which the gathering of the yeast is performed by a continuous mechanical operation.

A further object of my invention is to provide a process in which the separation of the yeast from the associated liquid and the drying of the yeast is accomplished in one operation.

A further object of my invention is to provide a process in which said liquid from which the yeast is separated may again be used in the propagation tank.

Other objects and advantages will appear in the following specification and the novel features of the invention will be particularly pointed out in the appended claims.

My invention is illustrated in the accompanying drawings forming part of this application, these drawings being a diagrammatic view of one form of apparatus for carrying out the invention.

The raw material upon which I operate consists of any clear sugared liquid. This may be a properly prepared and diluted molasses or glucose, or a totally saccharified clear grain must such as is now used in the ordinary process of manufacture.

For purposes of illustration, we will assume that the material to be operated on for the production of yeast is molasses. This is run into a boiler and diluting tank A where the molasses is first diluted, two gallons of water to one of molasses being a good proportion. The diluted molasses is then boiled with sulfuric acid. The boiling results in the conversion of the sucrose to glucose, and in the sterilization of the liquid, and the latter is pumped by means of the pump P from the tank A through the pipe *a* and into the filter B. Here the liquid is filtered and the greater portion passes by means of the pipe *b* into the second diluting tank C. Here the liquid is again diluted and is passed through the refrigerator R where it is cooled before passing into the propagation tank D.

Owing to the fact, as shown later, that there is a continuous aerating of the liquid during the entire process of fermentation, a temperature of approximately 30° C. is maintained in the propagation tank. In this propagation tank are added the necessary nutritive salts, such as potassium sulfate, ammonium phosphate, etc., in the amounts needed.

During the filling of the propagation tank with the sugared liquid the latter is run into the tank to approximately one-fifth of the capacity of the tank and then a portion of the pure yeast is run in from the pure yeast apparatus E by means of the pipe *e*. The sugared liquid continues to run in until say approximately half of the tank is filled and then another portion of the pure yeast is run in from the tank E and the final portion of the yeast is sent in to fill the tank up to the level X. While this is being carried on, and in fact throughout the entire process, pure air is passed into the bottom of the propagation tank through the pipe *d* which connects with a pipe *f* leading to the air purifier and compressor F. This same pipe *f* enters the boiler A and during the boiling and diluting of the molasses air is passed in, thus tending to hasten the operation through the agitation effected by the passage of the air. The pipe *f* also communicates with the bottoms of the tanks C and E and the air tends to agitate the liquids in these tanks besides aerating them.

Right here I wish to call attention to the fact that in continuously aerating the liquid in the tank D, I provide the necessary amount of oxygen for the maximum propagation of the yeast because the latter if de-

nied the oxygen from the purified air would get it from the sugar, thereby forming alcohol. The continuous aeration of the liquid in the propagation tank I regard as one of the main features of the invention, because it is due to the fact that the oxygen is supplied to the yeast in sufficient quantity from the air and not from the sugar that I am able to manufacture a much larger amount of yeast from a given quantity of raw material than can possibly be manufactured by other processes in which such continuous aeration is not practised and in which the sugar is largely converted to alcohol in the propagation of the yeast. Now the alcohol produced of course can be utilized, but it is of much lower value than the yeast and consequently the difference in values is a direct loss to the yeast manufacturer. Besides supplying all the air necessary to the yeast cells, the air also serves as a temperature controlling agency and permits the work to be carried on with settings of 8 to 10 Baumé. As fast as the yeast is formed it comes to the surface of the liquid in the tank D in the form of a scum or blanket and here it is continuously skimmed off by mechanical means. This mechanical means is indicated at *m* in the drawing, but it will be understood that the illustration is purely diagrammatic, the mechanical device for carrying out this process being the subject of a separate application.

The scum or froth which is skimmed from the tank D passes downwardly through the pipe *q* to either of the centrifugal separators 1 or 2, depending upon which of these separators is in operation. The valves *h'* and *k'* in the pipes *h* and *k*, respectively, control the flow of the froth or scum containing the yeast when either of these valves is open and the other is closed, and the scum or froth will flow into the centrifugal separator past the open valve.

The purpose of having two separators is to permit the continuous operation of the apparatus as a whole while removing the yeast from one of the separators. These centrifugal separators are of large size and can be run at a slow speed as well as at a high one. They are provided with an exterior shell or casing such as that shown at *s* and an interior rotary part *s'* having perforated sides against which is disposed a felt lining s^2. The space between the inner and outer portions communicates with a discharge pipe *p* which leads into a liquid receiving tank T. A cold water pipe *t* has branches which enter the centrifugals so as to wash the yeast on the interior walls of the felt lining. The liquid is thrown with the yeast against the felt lining, the liquid passing through the lining and also through the perforated wall *s'* and is delivered by the pipe *p* into the liquid receiving tank. The yeast is retained by the felt lining. When a certain amount of the liquid has been separated from the yeast then the valve v' is closed and the valve v^2 is open. Cold water is now turned in by the opening of the valve v^2 in the pipe *t* and flushes the yeast which is held to the felt lining. This water passes on through the perforated sides of the centrifugal separators and out through the pipe V as waste water. The liquid in the receiving tank T is pumped by means of a pump U through the pipe *u* and into the compensating tank W. This tank is provided with an automatic regulator (not shown) for maintaining a constant level of liquid in the propagation tank.

After the yeast in the centrifugal separator has been washed the separator is further rotated to dry the yeast. The drying is further attained by adding to the yeast about 10% of its weight of fine starch or potato fecula, so that the yeast will be in good condition to be discharged from the separator and also that it may be easily handled when it is subsequently compressed and made into compressed yeast cakes. During the removal of the yeast from one separator, as stated, the other separator is brought into operation. It will be noted that the rotation of the separators tends to aerate the yeast. Thus it will be observed that all along through the entire process I supply the yeast with air. This, as stated above, tends to a larger production of yeast than could possibly be obtained where the process does not afford facilities for supplying the amount of air necessary for the maximum propagation.

When all the yeast that can be procured by gathering the scum has been recovered the valve *y* in the pipe Y at the bottom of the propagation tank is opened, thereby passing the liquid of the propagation tank through the centrifugal separator so that any yeast which is in the liquid and which did not rise to the surface in the form of scum is centrifugated out and is recovered in the separators. Here again the process differs from the ordinary process in which yeast in suspension in the liquid of the fermentation tanks is lost for the yeast manufacture.

The foregoing description of the process will enable one to readily see the advantages to be derived. The highest results obtained by ordinary processes in which the scum is removed from the propagating tank by hand are 24 kilograms of compressed yeast and 24 liters of alcohol (calculated at 100%) per hundred kilograms of grain.

By the process which forms the subject of the present invention by a pure yeast culture of a suitable liquid with the maximum of sugar the product of "dry yeast" will be 20% of the total weight of the sugar or

100% of the total weight of the sugar when made into compressed yeast, that is to say, 100 pounds C. P. sugar, can produce at least its weight in compressed yeast.

By my process it is true very little alcohol is produced for the reasons stated, but the purpose of the invention is to increase the yield of yeast at the expense of alcohol, since the yeast is the more valuable of the two.

There is another difference between the present process and that which has heretofore been used. The empirical preparation of heavy musts consists in making a sufficiently mucilaginous and viscous must of such a nature that the carbonic acid gas cannot easily separate itself from the globule of yeast, and carries it along to the surface of the must where it is gathered by skimming it by hand with a scoop. The yeast cells which do not rise to the surface, those that remain in suspension in the must, or those that sink to the bottom of the tank are lost in the manufacture of the yeast. Besides this the method of skimming by hand while the must is in full fermentation, takes away from the must a large amount of liquid containing unfermented sugars, which are lost when the yeast is washed. On the other hand, by the present process all of the sugar is utilized for yeast by passing the separated liquid from the centrifugals back into the propagation tank and in subsequently passing the contents of the propagation tank through the centrifugals.

It will thus be seen that I have provided a process in which a number of advantages are attained. A process of continuous aeration of the yeast during the entire period of yeast production; a process in which the operation is continuous; a process in which the aeration takes place in the separator; a process in which the separating and drying constitute virtually one operation, and finally, a process in which the spent liquid is returned to the propagating tank for propagating the undeveloped values.

I have spoken of the return of the liquid separated from the yeast cells, into the compensating tank W from whence it subsequently passes into the propagation tank. This liquid, as has been explained, has certain undeveloped values. I may add to the liquid enough sugared solution so as to bring the density of the main body of the yeast propagating liquid in the propagation tank D to its original density, thus utilizing to the fullest extent the liquid thrown off by the centrifugals.

I claim:—

1. The herein described steps in a process of making yeast, which consists in setting a sugared liquid for yeast propagation while continuously aerating the liquid, continuously skimming off the froth or scum containing the yeast at the top of the liquid, and continuously separating the yeast cells from the liquid portion containing the non-spent sugar, and returning the latter to the main body of the sugared liquid.

2. The herein described steps in a process of making yeast, which consists in setting a sugared liquid for yeast propagation while continuously aerating the liquid, continuously skimming off the froth or scum containing the yeast at the top of the liquid, and continuously separating the yeast cells from the liquid portion containing the non-spent sugar, by centrifugation in the presence of pure air and returning the latter to the main body of the sugared liquid.

3. The herein described process of making yeast, which consists in setting a sugared liquid for yeast propagation while continuously aerating the liquid, continuously skimming off the froth or scum containing the yeast at the top of the liquid, continuously separating the yeast cells from the liquid portion while simultaneously aerating the yeast cells and said liquid portion during said separation and returning said separated liquid portions to the main yeast propagating body.

4. The herein described steps in a process of making yeast, which consists in setting a sugared liquid for yeast propagation, continuously skimming off the froth or scum containing the yeast at the top of the liquid, continuously separating the yeast cells from the liquid portions containing the non-spent sugar, continuously returning the latter to the main body portion of the sugared liquid and in continuously aerating the yeast during the skimming and during the separation of the yeast from the liquid portion.

5. The herein described process of making yeast, which consists in propagating pure yeast in a pure sugared liquid in the presence of nutritive salts and air, continuously skimming off the scum or froth containing the yeast cells at the top of the liquid, continuously separating the yeast cells from the liquid, aerating the liquid during the entire period of propagation, and returning the separated liquid portion to the main body of the yeast making material, whereby the undeveloped values in the liquid thus returned may be propagated and recovered.

6. The herein described process of making yeast, which consists in propagating pure yeast in a pure sugared liquid in the presence of nutritive salts and air, continuously skimming off the scum or froth containing the yeast cells at the top of the liquid, continuously separating the yeast cells from the liquid, aerating the liquid during the entire period of propagation, returning the separated liquid portion to the main body of the yeast making material.

whereby the undeveloped values in the liquid thus returned may be propagated and recovered, and in subsequently subjecting all of the liquid which forms the main body of the yeast propagating material to centrifugal action whereby the yeast in the liquid is recovered.

7. The herein described process of making yeast, which consists in propagating pure yeast in a pure sugared liquid of a given density in the presence of nutritive salts and air, separating the scum containing the yeast thus propagated from the main body of the liquid, subsequently separating the yeast cells from the associated liquid of the scum, adding sufficient sugared solution to the separated liquid to bring the main sugared solution to its original density when the separated liquid is returned to it.

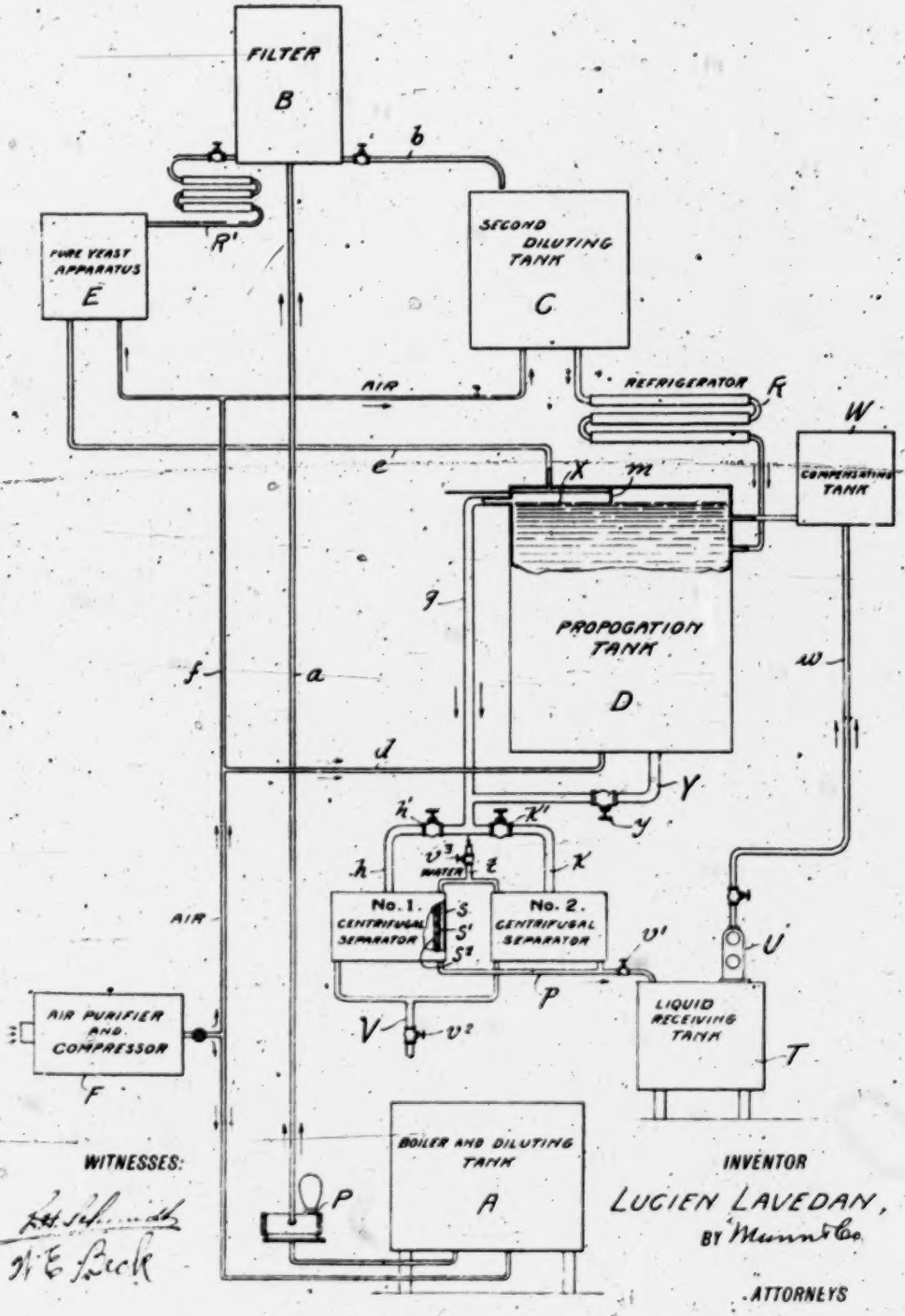
LUCIEN LAVEDAN.

L. LAVEDAN.

PROCESS OF MANUFACTURING YEAST.

APPLICATION FILED FEB. 12, 1916.

1,201,062. Patented Oct. 10, 1916.



WITNESSES:

H. E. Beck

INVENTOR

LUCIEN LAVEDAN,

BY Munn & Co.

ATTORNEYS

N° 20,787 A.D. 1892

Date of Application, 16th Nov., 1892
Complete Specification Left, 16th Aug., 1893—Accepted, 11th Nov., 1893

PROVISIONAL SPECIFICATION.

Improvements in the Manufacture of Yeast.

I, FLÉGONT MIKAÏLOVITCH POLSKY, of 65 Rue Emérian Paris in the Republic of France, Architect, do hereby declare the nature of this invention to be as follows:—

This invention relates to an improved manufacture of yeast without the production of alcohol.

The yeast employed in making bread and in distilleries is a product resulting from distillation in the same way that the yeast necessary for brewing beer is obtained during the brewing operation. It is unnecessary to describe here these two operations it being sufficient to note that in the actual processes only about one tenth part of the substance employed can be converted into yeast.

Knowing that yeast is much more costly quantity for quantity than alcohol or beer I have sought for and discovered a process by means of which I can convert nearly the whole of the substance employed into yeast without any portion being converted into alcohol.

In carrying out my invention I take a suitable quantity of green malt, or barley previously bruised, and the same quantity of flour prepared from wheat malt or from green or dry wheat malt, likewise bruised, and the same quantities of ordinary rye flour. I mix these ingredients together by adding thereto sufficient water heated to a temperature of from 50° to 60° C. to transform the substances employed into a paste which after carefully kneading I allow to remain for from 2½ to 3 hours in an open vessel to allow the substances employed to turn sour.

At the end of this time I add water at a temperature of from 90° to 95° C. as quickly as possible until the diluted mixture is of a temperature of from 62° to 65° C. I then cover the vessel and allow the mixture to remain for about an hour and a half to allow the farinaceous matters to be transformed into sugar.

On the mixture being uncovered I cool it as quickly as possible by plunging therein, for example in summer, vessels filled with cold water or ice, until the temperature falls to about 32° C. and I then add ordinary pressed yeast previously prepared in the following manner:—For the total of all the dry substances employed I take 10 *per cent.* of pressed yeast and after having diluted these two substances in water at 32° C. or in a portion of the mixture from the vessel the proportion being about four times more in weight than that of the yeast I allow the mixture to rest for about half an hour to allow the pressed yeast time to ferment. I then add for one gramme of pressed yeast 0·907 gramme or 0·908 gramme of tartaric acid ($C^4H^6O^6$); I then pour this mixture into the mixture in the vessel and about two hours afterwards I add the same quantity of bicarbonate of soda ($NaO, 2CO^2$) or carbonate of potash (KO, CO_2).

After adding the bicarbonate of soda I allow the mixture to remain for about four hours occasionally stirring it and during this time the process of the multiplication of the yeast commences.

Half an hour after having added to the mixture the yeast and substances above mentioned I commence to prepare a second mixture exactly in the same way as the first but taking only half the quantities of malts and flours and instead of adding thereto tartaric acid and bicarbonate of soda or carbonate of potash I add the same quantity of carbonate of ammonia (AzH^4Cl) or of sal ammoniac (H_4NCl) dissolved in a small quantity of water and poured into the fermented yeast in the same manner as described with reference to the first mixture.

[*Price 8d.*]

When the second mixture is ready I mix it with the first mixture and I then allow this fresh mixture to remain for from 6 to 8 hours to allow the yeast to form definitively after which the yeast is precipitated on the bottom of the vessel.

After passing the yeast through a sieve in order to separate it from the husks and other impurities, which I sometimes do before mixing the two mixtures together, I proceed to wash the yeast which is performed in a solution of phosphate of lime (Pho 3 CaO). I always prefer to employ the phosphate of lime throughout the process.

The pressing of the yeast does not differ from the processes at present in use and I do not therefore describe it.

By my improved process I can obtain a very white pressed yeast in the proportion of about 90 *per cent.* of the weight of the substance employed. If colour is not important, as it frequently is not, wheat flour need not be employed and the absence of this flour or of wheat malt greatly facilitates the process of multiplication of yeast. For the latter reason I sometimes dispense with the washing in the solution.

Dated this 16th day of November 1892.

G. F. REDFERN & Co.,
4, South Street, Finsbury, London, Agents for the Applicant.

COMPLETE SPECIFICATION.

Improvements in the Manufacture of Yeast.

I, FLÉGONT MIKAÏLOVITCH POLSKY, of 65, Rue Emériau, Paris, in the Republic of France, Architect, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to an improved manufacture of yeast without the production of alcohol.

The yeast employed in making bread and in distilleries is a product resulting from distillation in the same way that the yeast necessary for brewing beer is obtained during the brewing operation. It is unnecessary to describe here these two operations, it being sufficient to note that in the actual processes only about one tenth part of the substance employed can be converted into yeast.

Knowing that yeast is much more costly quantity for quantity than alcohol or beer I have sought for and discovered a process by means of which I can convert nearly the whole of the substance employed into yeast without any portion being converted into alcohol.

In carrying out my invention I take a suitable quantity of green malt, or barley previously bruised, and the same quantity of flour prepared from wheat malt or from green or dry wheat malt, likewise bruised, and the same quantities of ordinary rye flour. I mix these ingredients together and to one half the quantity I add sufficient water heated to a temperature of from 50° to 60° C. to transform the substances employed into a paste which after carefully kneading I allow to remain for from 2½ to 3 hours in an open vessel to allow the substances employed to turn sour.

At the end of this time I add water at a temperature of from 90° to 95° C. as quickly as possible until the diluted mixture is of a temperature of from 62° to 65° C. I then cover the vessel and allow the mixture to remain for about an hour and a half to allow the farinaceous matters to be transformed into sugar.

On the mixture being uncovered I cool it as quickly as possible by plunging therein, for example in summer, vessels filled with cold water or ice, until the temperature falls to about 32° C. and I then add ordinary pressed yeast previously prepared in the following manner:—For the total of all the dry substances employed I take 10 *per cent.* of pressed yeast and after having diluted this substance

in water at 32° C. or in a portion of the mixture from the vessel, the proportion being about four times more in weight of water than that of the yeast taken from the vessel, I allow the mixture to rest for about half an hour to allow the pressed yeast time to ferment. I then add for one gramme of pressed yeast 0·007 gramme or 0·008 gramme of tartaric acid ($C_4H_6O_6$): I then pour this mixture into the mixture in the vessel and about two hours afterwards I add the same quantity of bi-carbonate of soda ($NaHCO_3$) or carbonate of potash (K_2CO_3).

After adding the bi-carbonate of soda I allow the mixture to remain for about four hours occasionally stirring it and during this time the process of the multiplication of the yeast commences.

Half an hour after having added to the mixture the yeast and substances above mentioned I commence to prepare a mixture from the other half of the mixed ingredients exactly in the same way as the former mixture but instead of adding thereto tartaric acid and bi-carbonate of soda or carbonate of potash I add the same quantity of carbonate of ammonia $(NH_4)_2\,CO_3$ or of sal ammoniac (NH_4Cl) dissolved in a small quantity of water and poured into the fermented yeast in the same manner as described with reference to the first mixture.

When the second mixture is ready I mix it with the first mixture and I then allow this fresh mixture to remain for from 6 to 8 hours to allow the yeast to form definitively after which the yeast is precipitated on the bottom of the vessel.

After passing the yeast through a sieve in order to separate it from the husks and other impurities, which I sometimes do before mixing the two mixtures together, I proceed to wash the yeast which is performed in a solution of phosphate of lime ($Ca_3(PO_4)_2$). I always prefer to employ the phosphate of lime throughout the process.

The pressing of the yeast does not differ from the processes at present in use and I do not therefore describe it.

By my improved process I can obtain a very white pressed yeast in the proportion of about 90 *per cent.* of the weight of the substance employed. If colour is not important, as it frequently is not, wheat flour need not be employed and the absence of this flour or of wheat malt greatly facilitates the process of multiplication of the yeast. For the latter reason I sometimes dispense with the washing in the before mentioned solution of phosphate of lime.

My process enables me to obtain yeast by employing any kind of farinaceous cereals and other vegetables containing starch.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. A process for manufacturing yeast, preferably pressed yeast without the production of alcohol consisting essentially in taking a suitable quantity of green malt, bruised barley for example, and a like quantity of flour prepared from wheat malt or a like quantity of green or dry wheat malt likewise bruised, mixing these quantities of barley and malt flour or wheat malt, dividing the mixture into two equal parts and adding to each part water at 50 or 60 degrees C., kneading the paste so obtained and leaving it to rest in an open vessel to allow the substances to turn sour, adding water at 90 to 95 degrees C. in order to obtain a temperature of 62 to 65 degrees C., the vessel being then covered and the mixture left to rest to allow the farinaceous matter to be converted into sugar, quickly cooling the mixture until the temperature falls to about 32 degrees C., adding ordinary pressed yeast, diluting the mixture in water at 32 degrees C. and adding, after a period of rest, tartaric acid and bi-carbonate of soda or carbonate of potash; preparing in a similar manner a second mixture with the other half of the malts first referred to, but substituting carbonate of ammonia or sal ammoniac for the tartaric acid and bi-carbonate of soda or carbonate of potash, mixing the two mixtures, leaving the whole to rest for 6 to 8 hours, passing the yeast through a sieve, and washing and

pressing it by the ordinary methods, one or more washings being effected, if required, in a solution of phosphate of lime, the wheat flour being omitted if it is not required to produce a colourless yeast substantially as hereinbefore described.

2. As a new commercial product the pressed white or coloured yeast resulting from the process described without the production of alcohol.

Dated this 16th day of August 1893.

G. F. REDFERN & Co.,
4, South Street, Finsbury, London, Agents for the Applicant.

London: Printed for Her Majesty's Stationery Office, by Darling & Son, Ltd.—1893

N° 21,011 A.D. 1896

Date of Application, 22nd Sept., 1896
Complete Specification Left, 21st June, 1897—Accepted, 14th Aug., 1897

PROVISIONAL SPECIFICATION.

A New or Improved Method of and Apparatus for the Manufacture of Pure Yeasts.

I, GEORGE EUGÈNE JACQUEMIN, Chemist, of Malzéville, near Nancy, France, do hereby declare the nature of this invention to be as follows:—

The object of my invention is a new or important method of and apparatus for the manufacture of pure Yeasts.

The employment of pure wine Yeasts which I was the first to recommend five years ago in substitution for Yeast from grain or beer, is very advantageous, especially since the improvement which I have made in the manufacture of this Yeast rendering it by special culture more resistant to bacteria; my new method allows pure Yeasts to be used freely by reason of their continuous, convenient and rapid manufacture.

For the purpose of my invention, I employ a sterilizer supplied with molasses (preferably at the lower part) at about 28° Baumé, but any other fermentable sugared wort may be employed with the addition of other matters or ingredients useful for fermentation, as for example, Maltopeptone, Phosphate of Ammonia, *etc.*: and which are submitted to a long sterilization by steam clearing by any suitable means. This sterilizer may serve for saccharifying the grain for the preparation of a thick Yeast.

When the sterilizing operation is complete the steam inlet is shut off and air is forced in by an air compressor, and an outlet cock at the upper part of the apparatus is opened, the sterilizer being cooled at the same time which may be easily effected by a sheet of water flowing down its sides.

When the wort thus treated is at a temperature of 28° to 30° C. it is conducted by a pipe into a vessel for producing the first Yeast.

The quantity of wort usually introduced is about a hectolitre; the selected pure Yeast is introduced at the Thermometer opening, so that the interior of the vessel should be as little as possible in contact with the exterior atmosphere. The Thermometer is then replaced and the Yeast is aërated during the fermentation (revived) of the Yeast; when this takes place and the fermentation is well in activity this first Yeast is passed into a receiver of a larger capacity and sterilized wort from the sterilizer is poured on this Yeast, which results in a new and very important multiplication of Yeast. These substances or materials are then aërated during about 12 hours and a certain quantity is then drawn off into a vat, and which is charged with wort. In this way, a third-Yeast is obtained which as soon as made is directed, according to the requirements of the manufacture, into fermenting vats.

The quantity of Yeast taken from the receiver is replaced by an equal quantity of sterilized wort from the sterilizer, and so on each three, four, five, or six hours depending on the number of vats to be fermented during 24 hours or longer and according to whether the Yeasts remain pure, or commence to be contaminated.

[*Price 8d.*]

If contamination is produced the operations are recommenced with pure Yeast as explained above.

In my arrangement of apparatus, I connect the air conduits and the wort conduits to the steam pipe, so that the sterilizing can proceed when necessary.

In the same way the air forced by the compressor into the air reservoir will be sterilized in passing through one or more filters with filtering media such as antiseptic wadding &c. The sterilizing of the air may be completed by washing it in antiseptic solutions.

I do not limit myself to the manufacture of pure wine Yeasts for which my method is particularly advantageous, and it is understood that my method and arrangement of apparatus may be applied to the continuous production of any other kind of Yeasts.

I may also replace the aëration by the introduction of Oxygen which may be effected by means of tubes containing this gas in a compressed state as sold commercially, thus avoiding a special installation for the purpose.

I may also dispense entirely with any aëration or oxygenation for the production of pure Yeasts, my experience proving that this may be done by employing a nourishing wort richer in matters assimilable by the Yeast; this last mode of operation is more costly but where for certain cases or reasons, the aëration or oxygenation cannot be employed, it is capable of rendering useful service.

Dated this 22nd day of September 1896.

H. GARDNER,
166 Fleet Street, London, Patent Agent,
Agent for the said George Eugène Jacquemin.

COMPLETE SPECIFICATION.

A New or Improved Method of and Apparatus for the Manufacture of Pure Yeasts.

I, GEORGE EUGÈNE JACQUEMIN, Chemist, of Malzéville, near Nancy, France, do hereby declare the nature of this invention and in what manner the same is to be performed to be particularly described and ascertained in and by the following statement:—

The object of my invention is a new or improved method of and apparatus for the manufacture of pure Yeasts.

The employment of pure Wine Yeasts, which I was the first to recommend five years ago in substitution for Yeast from Grain or Beer, is very advantageous, especially since the improvement which I have made in the manufacture of this Yeast rendering it by special culture, more resistant to bacteria; my new method allows pure Yeasts to be used freely by reason of their continuous, convenient, and rapid manufacture.

In the annexed drawings, I show (as an example) the general arrangement of apparatus for this manufacture, but the details and accessories, pipes &c. may be modified without altering the principle of my invention.

A, is a sterilizing receiver of the worts. B, is a receiver for mother Yeast (levure mère). C, is a receiver for developing the Yeast. D, is a preparation vat. E, is a fermentation vat. F, is an air compressing apparatus. G, is a compressed air reservoir. H, is an air filter. I, I, are the conduits for the worts, and I', I', the conduits for the sterilized worts. J, J, the steam pipes. K, K, the air pipes.

The receiver A is a sterilizer supplied with all the necessary adjuncts, it can be cooled in any convenient manner, the molasses which I use for my process is placed in it (preferably at the lower part of the receiver) at about 28° Baumé, but any other fermentable sugared wort may be employed with the addition of other matters or ingredients useful for fermentation, as for example:—Maltopeptone, Phosphate of Ammonia, *etc.*: and which are submitted to a long sterilization by steam. This sterilizer may serve for saccharifying the Grain for the preparation of a thick Yeast.

When the sterilizing operation is complete, the steam inlet is shut off and air is forced in by an air compressor, such as at F, and an outlet cock at the upper part of the apparatus is opened, the sterilizer being cooled at the same time, which may be easily effected by a sheet of water flowing down its sides.

When the wort thus treated is at a temperature of 28° to 30° C. it is conducted by a pipe I^1 into a vessel B which is a receiver for producing the first Yeast. This vessel B should be previously sterilized.

The quantity of wort usually introduced is about a hectolitre; the selected pure Yeast is introduced at the Thermometer opening, so that the interior of the vessel or receiver should be as little as possible in contact with the exterior atmosphere. The Thermometer is then replaced and the Yeast is aërated for the reviving fermentation; when this takes place and the fermentation is well in activity, this first Yeast is passed into another receiver C of a larger capacity, and sterilized wort from the sterilizer A is poured on this Yeast, which results in a new and very important multiplication of Yeast. These substances or materials are then aërated during about 12 hours, and a certain quantity is then drawn off by the cock P into a vat D standing on legs and which is then charged with wort. In this way, a third Yeast is obtained which as soon as made is directed according to the requirements of the manufacture into the fermentation vats E.

The quantity of Yeast taken from the receiver C is replaced by an equal quantity of sterilized wort from the sterilizer A, and so on for each three, four, five, or six hours depending on the number of vats to be fermented during 24 hours or longer, and according to whether the Yeasts remain pure, or commence to be contaminated.

If contamination is produced, the operations are recommenced with pure Yeast, as explained above.

In my arrangement of apparatus, I connect the air conduits K and the wort conduits I^1 to the steam pipe J, so that they can be easily sterilized when necessary.

In the same way, the air forced by the compressor F into the air reservoir G will be sterilized in passing through one or more suitable receivers H provided with filtering substances such as antiseptic wadding &c. The sterilizing of the air may be completed by washing it in antiseptic solutions but this is not indispensable.

As before stated, I do not limit myself strictly to the manufacture of pure Wine Yeasts, for which my method is particularly advantageous, and it is understood that my method and arrangement of apparatus may be applied to the continuous production of any other kind of Yeasts.

I may also replace the aëration by the introduction of oxygen which may be effected by means of tubes containing this gas in a compressed state as sold commercially, thus avoiding a special installation for the purpose.

I may also dispense entirely with any aëration or oxygenation for the production of pure Yeasts, my experience proving that this may be done by employing a nourishing wort richer in matters assimilable by the Yeast; this last mode of operation is more costly, but where for certain cases or reasons the aëration or oxygenation cannot be employed, it is capable of rendering useful service.

Having now particularly described and ascertained the nature of this invention and in what manner the same is to be performed I declare that what I claim is:—

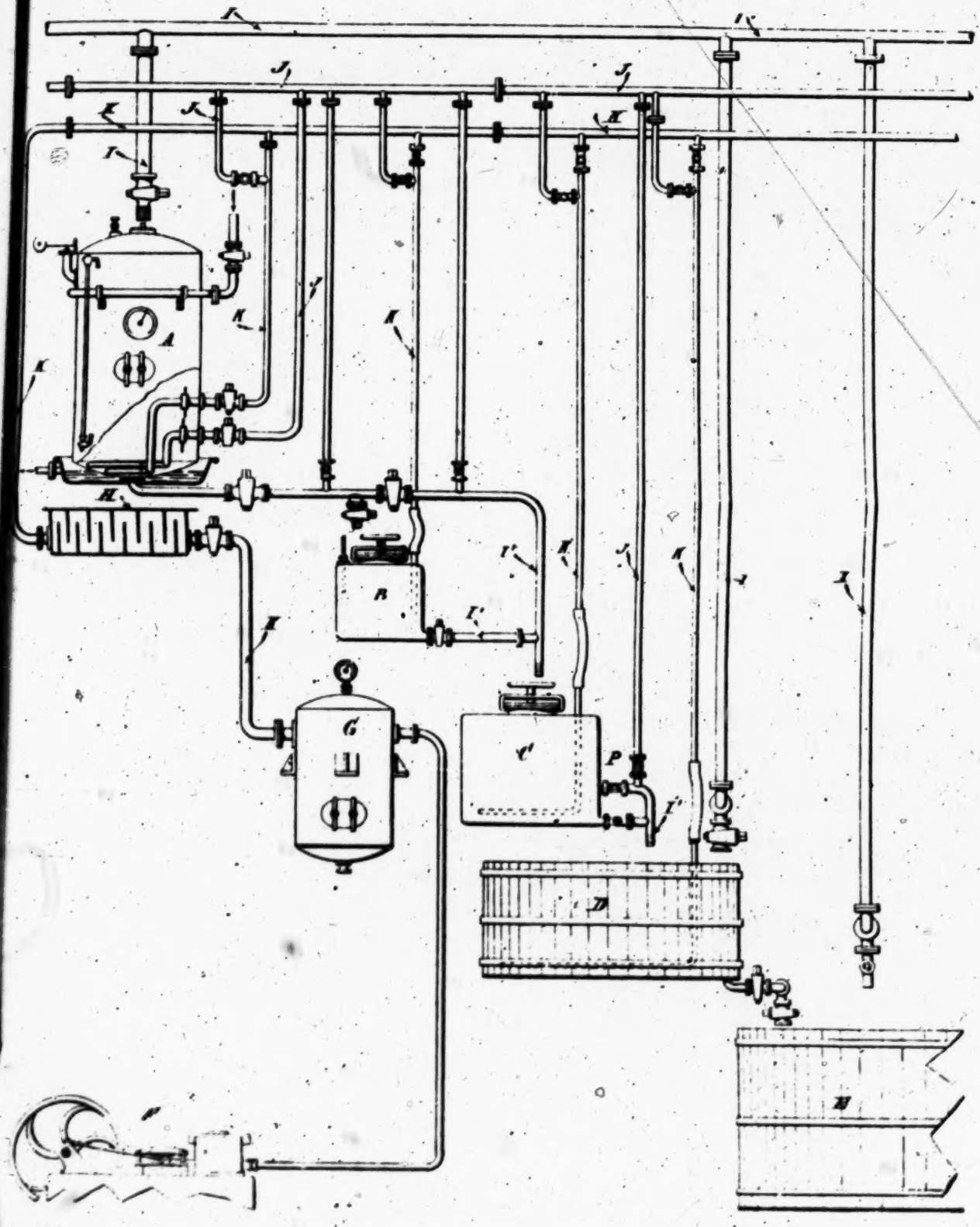
The new method of continuous manufacture of pure Yeasts above described, and

the employment of the arrangement of apparatus shown as an example on the annexed drawing.

The 21st day of June 1897.

H. GARDNER,
166 Fleet Street, London, Patent Agent,
Agent for the said George Eugène Jacquemin.

Redhill: Printed for Her Majesty's Stationery Office, by Malcomson & Co., Ltd.—1897.


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N° 11,617 A.D. 1913

(*Under International Convention.*)

Date claimed for Patent under Patents and Designs Act, 1907, being date of first Foreign Application (in France), 25th May, 1912

Date of Application (in the United Kingdom), 19th May, 1913

At the expiration of twelve months from the date of the first Foreign Application, the provision of Section 91 (3) (a) of the Patents and Designs Act, 1907, as to inspection of Specification, became operative

Accepted, 30th Apr., 1914

COMPLETE SPECIFICATION.

Improvements in Yeast or Ferments for use in Fermentation and Distillation Processes.

I, ALFRED MOLIHANT, of The Hainaut Institute of Hygiene and Bacteriology, Mons, Belgium, Chemist and Engineer, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention has reference to improvements in yeast or ferments for use in fermentation and distillation processes and is intended more particularly for bringing about the fermentation of molasses or other fermentable liquids under such conditions that the wash constituting the residue of their distillation may be evaporated without incrusting the tubular apparatus which is employed for this purpose.

It is known that the usual operation for sulphuric acidification of mash or wort, particularly when the liquids are charged with earthy alkaline salts, brings about incrustation which interferes with the normal working; on the other hand, hydrochloric acidification obviates that difficulty but necessitates the employment of considerable quantities of hydrochloric acid, and this occasions very serious inconvenience on several grounds but more particularly because the acid rapidly attacks the whole of the apparatus; finally, working with hydrochloric acid in the usual way necessitates strong acidity in hydrochloric acid to obviate the overrunning of the liquid in fermentation by numerous bacteria which diminish considerably the output of alcohol.

In order to attain the object aimed at, *i.e.* the obviation of any incrustation of the evaporating apparatus, working with hydrochloric acid would be perfect if the operation could be carried out with very small doses of acid and if microbic infection could be prevented in these conditions. I have found as the result of many experiments that a yeast or ferment of a pure or composite stock acclimatised to hydrochloric acid, added together with the liquid in which it is cultivated and preserved, and which it has caused to ferment, to molasses or other alkaline fermentable liquid which has been diluted with water so as to obtain a reaction almost neutral to litmus, so that the mixture is only slightly acid, enables the fermentation of sugar juices to be carried right to the end

without bringing about infection. This result can only be obtained if the ferment which is forced to live in a medium acidified by hydrochloric acid has been itself accustomed to increasing doses of formic aldehyde. A ferment acclimatised by preliminary cultures in a formolated medium until the critical dose is arrived at, then transferred into a hydrochloric medium, and thence into an almost neutral medium, such as crude molasses diluted and only slightly alkaline, has the interesting property that it offers a considerable resistance to bacterial invasion.

A suitable liquid in which to sow and nourish the yeast is a solution of 70 grammes of sugar candy, 4 gr. each of tartaric acid and nitrate of ammonium, 0·6 gr. each of phosphate of ammonium and carbonate of potassium, 0·4 gr. of carbonate of magnesium, 0·25 gr. of sulphate of ammonium, and 0·07 gr. of silicate of potash in 1500 gr. of water with the addition of 0·2 to 0·3 gr. of formic aldehyde per litre. The yeast grown in this medium is placed together with the said medium in propagating vats and is developed and acclimatised to hydrochloric acid in molasses diluted with water and acidified by the addition to each litre of 3·5 gr. of commercial hydrochloric acid of 20° Bé., the vats being filled up gradually, and then crude molasses is added in the proportion of about 2 to 1, the proportion being varied according to the nature of the molasses and the working conditions.

The process described obviates all the difficulties referred to. Mash or wort fermented with a yeast or ferment that has been subjected to the treatment described yields a wash that can be concentrated without any difficulty. My process has other advantages in that it obviates the denitration and sterilisation necessary in the process hitherto followed.

Having now particularly described and ascertained the nature of my said invention, and in what manner the same is to be performed, I declare that what I claim is:—

1. In processes of fermentation of molasses and other fermentable liquids, the employment of yeast or ferment of a pure or a composite stock acclimatised to hydrochloric acid after having been previously accustomed to live in formolated mediums.

2. Yeast or ferment for use in the fermentation of molasses and other fermentable liquids, acclimatised to live in a medium acidified by hydrochloric acid after having been previously accustomed to increasing doses of formic aldehyde.

Dated this 19th day of May, 1913.

BAUER, IMRIE & SLACK.
Chartered Patent Agents & Consulting Engineers,
72—74, Cannon Street, London, E.C.

Redhill: Printed for His Majesty's Stationery Office, by Love & Malcomson, Ltd.—1914.

GERMAN PATENT NO. 10135

PATENT SPECIFICATION

1879 No. 10135 Class 6.

DR. JACQUES RAINER in VIENNA

PROCESS OF MANUFACTURING PRESSED YEAST WITHOUT ALCOHOLIC FERMENTATION AND WITHOUT THE FORMATION OF BY-PRODUCTS.

Patented in Germany, from Oct. 12, 1879 on.

My invention is based on the ~~discovery~~ experience derived from intensive studies and experiments that the culture (growing) of the yeast plant (Sacharomices cerevisiæ) can take place in suitable media not only without fermentation, but that also the cells formed by a simple process of vegetation, in other words the application of the above principle, can be utilized for the ~~commercial~~ industrial production of pressed yeast.

The procedure for the commercial production of the new pressed yeast is as follows:

The vegetable proteins derivable from cereals, corn, legumes, or industrial offals, such as bran, malt ~~germs~~ sprouts, spent grains, gluten, etc., are extracted with from 15 to 20 times their weight of slightly alkaline water and peptonized with lactic acid, added in excess (about 4%), or with mineral acids (phosphoric acid, about ¼%, sulphuric or hydrochloric acid, about 4/10%) at a temperature of from 30 to 32° R., or they ~~can~~ may also be immediately macerated with the said acids in weak solutions and at the same time converted into peptones. (In this connection it is to be stated that a portion of the proteins contained in dry malt ~~germs~~ sprouts,

viz. from 5 to 10% of the aggregate weight, is already peptonized by the preceding process of vegetation.)

The peptonization of the proteins is effected in the case of cereals, corn or legumes, as also in the case of brans or gluten, through an addition of diastase for which purpose the addition of one part by weight of sprouts dry malt or five parts by weight of malt ~~germs~~ for every part by weight of the dry weight of the proteins is amply sufficient; as mentioned before, the liquid in which the proteins are to be converted into peptones must contain lactic acid (about 4%), phosphoric acid (up to ¼%), sulfuric or hydrochloric acid (about 2/5%), as the presence of these acids is absolutely necessary for the process of peptonization.

The most suitable temperature for the peptonization is about 30° R., and a period of from 18 to 20 hours is sufficient for bringing it about, but nearly perfect results may ~~can~~ also be attained at lower temperatures and within a proportionally longer period of time.

In working up malt sprouts ~~germs~~ alone, an addition of malt is superfluous, the diastase contained therein being more than sufficient for the process of peptonization, so that the only requirement is the utilization of one of the acids mentioned above in the stated proportions.

The mixing of the malt sprouts ~~germs~~ with water can be carried out by hand or by means of a mechanically driven agitator. When preparing a mash of malt or cereals, in other words when preparing the required maltose, dextrose, etc., I use the usual factory equipment while applying the commonly used temperatures and concentration ratios.

Let it be further stated that due to the diastase in conjunction with acids even the mucilaginous pectates contained both in the malt ~~germs~~ sprouts and in other materials are liquefied, and when one is concerned with the preparation of pure peptones, these pectates can be separated by an endosmotic apparatus or dialyzer, in such a manner that the peptones dialyze in water through suitable membranes, whilst the gelatinous pectates remain behind as a residue.

The neutralization of the acids is effected by means of soda or by saturating the liquid with basic calcium phosphate, as about to be described.

The finished peptonic liquid, either with or without sugar content can be evaporated in a water or vapor bath, or, better still, in a vacuum to the consistency of a syrup or extract, or even to a dry condition, so that it ~~can~~ may be used in this state as a material fit for transportation.

The peptone-containing liquor is separated from solid matter (carbohydrates, vegetable fibres, etc.) by a simple lixiviation (maceration) or by pressing or centrifugation and in any event is carefully purified besides by filtration or sedimentation. Prior to purifying it by filtration or settling, it is advisable, however, to neutralize the acid present therein by means of soda or to saturate the liquid with basic calcium phosphate, the latter method being preferable as in this way the yeast is amply supplied with the necessary phosphoric acid.

Depending upon the peptone content of the liquor dextrinized starch which was converted into dextrose in the known manner by boiling it in the presence of mineral acids in an amount equal to about one-half of

the dry weight of the peptone, or maltose, molasses, or finally, sugar with a small amount of inoculating yeast (beer yeast or pressed yeast), is added to start the yeast growth, the mentioned carbohydrates, however, being always limited to no more than from ½ to 1% of the weight of the total liquor and being exclusively used for the formation of cellular tissue.

The vegatation of the yeast will take place in the best and most reliable manner at temperatures of from 14 to 17° R., as in the case of higher temperatures and also, when a greater amount of carbohydrates is added, losses are very apt to ensue due to the fact that the varieties of sugar used are partly converted into lactic acid or undergo alcoholic fermentation instead of merely supplying the cellular substance of the yeast.

The cultivation of yeast is carried out, as is customary in Holland, either in flat vessels (level of liquid about 14 cm high) in order to afford access of a sufficient amount of atmospheric air, or in a better and safer way, in fairly deep tubs made of wood, glass, cemented masonry or any other suitable material, into which atmospheric air is injected through hose or piping by means of mechanical force, by means of blowers or compression pumps, and in as finely divided a condition as possible as by means of sprinklers or similar devices.

It is even more advantageous to use instead of atmospheric air an air of increased ozone content or ozonized oxygen. The later is produced by successive additions of hydrogen peroxide to the nutrient solution, whereas the ozonization of the atmospheric air is brought about by means of phosphorous or by causing the atmospheric air to pass through a sealed vessel containing potassium permanganate and the necessary mineral acid and only then passing the air enriched with ozone into the nutrient solution.

According to the greater or lesser density of the nutrient liquor used, and according to the higher or lower temperature thereof, and also depending upon the amount of ozonized air that is introduced, the growth of the yeast after each substantial addition of dextrose, maltose, etc., is completed within from 6 to 8 hours. The peptone content of the mass may amount to 1-2% of the weight thereof, and even more, whereas in each instance only ½-1% of dextrose or other carbohydrate shall be added in order to suppress lactic acid or alcoholic fermentation with certainty, and thus also to avoid any losses in my process caused thereby.

When after a period of from 6 to 8 hours the added dextrose or other variety of sugar has been consumed entirely or at least the greatest part thereof for the growth of the yeast, an additional portion containing from 1/20 to 1/10% is added and likewise the peptones after having been consumed can be added again to the liquor, either in portions or in a slow and continuous stream, so that the same nutrient solution can with successive replacement of the consumed substances remain in use for entire weeks or months, unless it has been contaminated by other substances or secondary ferments and thus made unfit for further use.

In proportion as the materials required for yeast culture are added in portions, the yeast produced likewise is withdrawn successively and only the yeast which is still suspended in the liquor itself remains behind as ~~an inoculant~~ seed for the alcoholic ferments still to be formed.

Recovery of the yeast is effected as follows: It is skimmed off from the surface of the liquor, or after drawing off the latter it is taken from the bottom of the vessels upon which it has settled out as a fairly compact layer. In the latter case, it is advisable for

more extensive operations to mount the vessels in tier batteries, so as to permit of transferring the nutrient solution from one into the other.

In order to produce a yeast as pure as possible and free from secondary ferments, the nutrient solution can also be prepared in a thinner form ~~of~~, ~~as the example case may be~~, with a peptone content of from only ¾ to 1%, and the carbohydrates (dextrose, maltose, etc.) can likewise be added in smaller amounts (as the first dose about 1/3%, then ~~in~~ 3 to 3 hours about 1/20%), in which case the available peptones will be converted into yeast within from 12 to 15 hours at a temperature of from 16 to 17° R., provided a sufficient amount of pure air, possibly passed through sulphuric acid, or ozonized oxygen, is supplied. The whole liquor is then cooled by means of a refrigerator or through the addition of cold water or ice, if possible to 6-8° R., and the yeast obtained will settle out almost completely within from 12 to 14 hours on the bottom of the vessel from a height of liquid of from 80 to 90 cm., whereupon the spent nutrient solution can be drained off into the sewer.

The ~~steps of~~ purifying and pressing of the yeast prepared in accordance with my new process is carried out in the known manner, but in order to give it better keeping properties calcium phosphate can even prior to compression be added thereto in an amount corresponding to 5-10% of the total weight of the yeast to be finished.

According to results obtained up to the present, from 250 to 300 kg of a pure and vigorous pressed yeast can be obtained from every 100 kg of pure peptone (dry weight), only about 200 kg of dextrose or sugar being required for its cultivation.

PATENT CLAIMS

1. The culture of yeast cells from vegetable peptones with small additions of dextrose, maltose, or similar carbohydrates, which latter, however, are used exclusively for building up the cell walls.

2. The successive admission of peptones and carbohydrates into the relatively stable nutrient solution intended for yeast culture, and likewise, the successive withdrawal of the finished yeast, both in the proportions stated.

3. The process, based on the foregoing, of producing pressed yeast on an industrial scale, with exclusion of the customary alcoholic fermentation and without any by-product in the form of alcohol or vinegar.

GERMAN PATENT OFFICE

PATENT SPECIFICATION

No. 269,192.

Class 6a.

Group 16.

ISSUED JANUARY 12, 1914.

ADAM KAMIENSKI in DRESDEN.

PROCESS OF PRODUCING COMPRESSED YEAST FROM WORT BY THE AERATION PROCESS.

Date of patent in the German Empire:
September 20, 1912.

According to the new process the mashing and clarification may be carried out as desired according to known methods of working. Of the strong first wort draining from the clarifying vat of about 10 to 12° Balling one-third is held back in a vessel which is arranged above the fermenting vat, while the other two-thirds of the first wort drain into the fermenting vat. When these two-thirds of the first wort have been diluted by continued spargings to 8° Balling, two-thirds of the entire quantity of pitching yeast (which represent 8% of the mash materials) are added. Thereupon at a fermenting temperature of 23° R. (28.7° C.) slight aeration is started, and the wort in the fermenting vat is so far diluted by spargings that its saccharometer reading amounts to 3° Balling.

Air is then introduced in full amount and the fermenting temperature is lowered to 20° R. (25° C). The vat content then is given a chance to rise to 22° R. (27.5° C), the fermenting temperature is again de-

pressed to 20° R. (25° C.) and this raising of the temperature and subsequent depression will be repeated until the sacchorometer reads 1.8° Balling. Now the held back one-third of the first wort to which the retained one-third of the pitching yeast was added beforehand, is run to the fermenting vat. The fermenting temperature which now rises strongly is lowered to 20-22° R. (25-27.5° C.) and this temperature is maintained until the wort has been fermented to 1.2° Balling. At 1.2° Balling the contents of the vat are once more allowed to reach 24° R. (30° C.) and the yeast is separated from the wort in the customary manner when the latter has been fermented down to 0.7-0.8° Balling.

It is true that in the wort aerating process the wort at times is diluted to 3° Balling and then fermented, but in such cases the fermenting temperature is maintained at 17-18° R. (21-25.5° C). The wort in this process is strongly utilized and the yield of yeast is increased. On the other hand, fermentation at the lowered fermenting temperature requires relatively much time. Owing to the high fermenting temperature of the new process at the same time a reduction of the fermenting period is gained. In order, however, to prevent too fast fermentation, the fermenting temperature is periodically lowered which process is also known as such in the aeration yeast practice. The advantage of fermentation of highly diluted wort at a high fermenting temperature, as used according to the present process is based in opposition to fermentation of highly diluted wort at a fermenting temperature maintained on a low level on the fact that not only an increase of the yeast yield, but at the same time a reduction of the fermenting period is brought about.

Naturally when the fermenting temperature is maintained on a relatively high level of at least 20° R. (25° C) less advantage is taken of the raw material than in the case of slow fermentation at a low ferment-

ing temperature. As a compromise the pitching yeast is added in two stages together with the addition of the retained first wort. In this manner new nourishment is not only brought to the already fermenting fluid, but also, by means of the newly arriving pitching yeast further fermentation is introduced, in which the nutritive substances of the liquid are further utilized. Advantage is also taken of the material by the fact that the new vigorously beginning fermentation is going on in a wort which still is relatively thin.

There is likely to be occasionally in the process of yeast manufacture a second seeding with pitching yeast, but until now the latter was distributed in water and admixed to the already fermented liquid. But by this means a but insignificant new fermentation is brought about, because the already fermented liquid naturally offers little new nourishment to the newly arriving pitching yeast. The fundamental and novel idea connected with the present process is that the second seeding of the pitching yeast is performed before the fermentation is completed while concentrated wort is added.

While with the use of the well known yeast aeration process aeration as a rule requires a period of 12 hours, under the new process an aeration period of only 8 hours is needed, and in this manner a yield of from 36 to 40 percent of unobjectionable yeast is secured which is noted for keeping quality.

GERMAN PATENT SPECIFICATION No. 269,192

PATENT CLAIM

Process of producing compressed yeast from wort according to the aeration process alternately causing the fermenting temperature to rise and to lower, characterized in that wort and pitching yeast are successively used in two parts in a manner such that in the

first place two-thirds of the first wort diluted by spargings to about 8° B. are set to ferment with two-thirds of the pitching yeast; then the wort is diluted in the fermenting tub by sweet-water to about 3° B. and fermented to about 1.8° B. whereupon the held back one-third of the pitching yeast which is partly fermented in the retained one-third of the first wort is admixed to said fermented wort and the whole mass is fermented in the known manner.

ORIGINAL GERMAN PATENT NO. 269,192.

(Physical Exhibit.)

FRANCE

NATIONAL OFFICE OF INDUSTRIAL PROPERTY

PATENT No. 470,925

II—Alimentation

5.—Beverages, wines, vinegar, cooperage

PROCESS FOR OBTAINING MOLASSES YEAST.

Mr. Gustav Roth, residing in Austria.

Applied for March 30, 1914, at 3:47 P. M. in Paris.

Delivered June 27, 1914—Published Oct. 6, 1914.

(2 patent applications filed in Austria on April 2, 1913 and March 12, 1914—Statement of the applicant).

The treatment of molasses for manufacturing yeast presents, as is known, certain difficulties, especially in warm weather. It is particularly in regard to the regularity of quality that the product is not satisfactory. Sometimes the yeast is in good condition, but frequently one sees it assume a bad color accompanied by infections. Besides these drawbacks which become apparent when using the molasses for yeast making in general, there are, as is known, molasses of different origins and not all of them are suitable in an equal degree for the manufacture of yeast, with the present-day processes.

The present invention gives the solution of the problem consisting in obtaining in a reliable way with molasses a yeast of good quality, and that with or without intervention of lactic acid.

Ordinarily, in order to obtain the must by employing mineral acids exclusively (that is to say without production of lactic acid by the bacterial method) one proceeds as follows: After having diluted the molasses with water it is neutralized by mineral acids (sulphuric acid or hydrochloric acid) or, to speak more precisely,

there is added to it a small excess of acid so that the degree of acid represents 1.5 per 100 cm^3 of the mixture; it is heated by steam for a sufficient length of time in order that the molasses, freed from a dark slime which has settled during the cooling, yield a sufficiently clear product, which contains all its sugar in the state of dextrose. The clear liquid is placed in the maceration vat, then, after it has been brought to the desired degree of concentration and of acidity, there are mixed with it malt ~~germs~~ sprouts and it is passed into the clarification vat.

When one has recourse to the production of lactic acid by the bacterial method, the operation usually carried out for obtaining the clear must is the following: The molasses having been, as stated above, clarified by acidification by means of mineral acids and by boiling, the malt ~~germs~~ sprouts are wetted down and acidification is allowed to proceed in the vat. (The operating of mashing the ~~germs~~ sprouts can, however, be effected after the acidification). In order to bring about the acidification lactic acid bacteria are sown by means of a small starter of dry malt and rye, or by means of a certain quantity of a sufficiently acid must (coming from a preceding operation). When the must has attained the desired degree of acidity, it is sterilized by heating it up to 75° whereupon it is poured into the clarification vat.

After long tests it has been found that impurities still exist in the solution of molasses prepared and acidified in the ordinary way, then drawn off clear after boiling. These impurities are set free in very fine suspension when the molasses has been diluted a second time, acid added thereto, and is vigorously stirred or aerated. If the solution is then subjected to filtration by which it is freed from these very fine impurities, one obtains a clear liquid from which one gets regularly a

yeast of excellent quality. Another advantage of the new process is that the solution can be run directly into the fermentation vat in which there can be added clear extracts which contain the nitrogenous elements of the must. The filtration of the must becomes superfluous by the fact of inserting in accordance with the new process between the successive operations at present carried out, a second purification of the already clarified molasses solution. This results not only in a great simplification of the operations, but which is more important, one also avoids the troubles due to the fact that with the present method of filtration of the must prior to fermentation, the malt ~~germs~~ sprouts retain, more or less the very fine impurities still existing in the molasses, in spite of its clear appearance; now these impurities, as has been shown by precise experiments, injure considerably the quality of the yeast and consequently have a vital effect on this quality.

When one has recourse to the formation of lactic acid, the molasses solution purified in accordance with the new process, is treated with nitrogenous clear extracts, such as vinasse, nutritive or other salts, after having added thereto a powerful lactic acid ferment in the form of clear must, acidification is allowed to proceed for some hours, after which it is run off into the fermentation vat. In this case, too, the must is not filtered. It is of course understood that one could also proceed in such a way that a part of the lactic acid or even all the lactic acid will be produced prior to the filtration of the molasses.

Here are some examples of application of the process:

The molasses diluted to 35° Balling is treated with sulphuric acid in the proportion of 4 cm^3 per 100 cm^3 of the mixture, whereupon according to its quality it is subjected to a boil lasting several hours, after which

it is allowed to clear up. The clarified solution is drawn off, again diluted to 20° Balling, and sulphuric acid is added to it so that the degree of acid is the equivalent of 6 to 10 cm^3 per 100 cm^3. It is advisable then to aerate it for a certain time. The impurities are precipitated in a very finely divided form and are eliminated by a cautiously conducted filtration.

When one makes use of mineral salts exclusively, the purified molasses is placed directly into the fermentation vat and is made to ferment with clear nitrogenous extracts, vinasse, nutritive salts, etc. When one makes use at the same time of lactic acid of fermentation, the purified molasses solution that has become clear, is mixed in a vat with nitrogenous extracts, vinasse and nutritive salts, one dilutes it to 14° Balling and after inoculating it with clear must at 50° or 60°, ~~clear must~~ acidification is allowed to proceed. When the desired degree of acidity is obtained, it is set to ferment directly.

For the success of the preparation it is important to filter carefully so as to clear the molasses from the very fine impurities which are in suspension. It is advantageous to allow the filtration to take place without over-pressure and to give to the molasses a large surface of contact with the filter.

The impurities in suspension are in such a state of division that they pass through the closest filter when filtration is effected under pressure. Under these conditions the process becomes less efficacious or even is no longer efficacious at all. By allowing filtration to proceed slowly, without pressure, one is sure of avoiding the drawback pointed out above. In order to offset the slow rate of filtration when it is carried out without pressure, it is well to employ large filtering surfaces.

A filter meeting these requirements is the Kasalovsky filter.

After precipitating the suspended matter, the molasses is run directly into the Kasalovsky filter. The liquid passes through the surface of the filter while leaving the solid substances in the different compartments and comes out of the latter at the upper end. When one of these compartments, each of which operates independently, yields by accident a badly filtered liquid, the corresponding valve is closed. For greater safety a second filter is added or all the liquid is run a second time through the filter which holds back the last impurities.

Filters meeting the requirements demanded in the present case (the Kasalovsky filter for example) are already being used in the sugar industry, but their function then is to retain the lime coming from the clarification and saturation. In the manufacture of molasses yeast, the impurities are elements which existed in the molasses in the colloidal state and which must be eliminated in order to supply a pure and sound nutritive substance to the yeast. A few centimeters of substances in suspension in the must would be sufficient to spoil all the yeast obtained from the must.

RESUME

1. This process for preparing molasses yeast is characterized by the fact that the molasses solution, diluted and acidified in the ordinary way and drawn off clear after boiling, is diluted again and acidified so that by agitating or aerating the solution, the very fine substances in suspension separate, and that the precipitated impurities are eliminated by filtration.

2. One mode of carrying the process into practice while using mineral acids exclusively, is characterized by the fact that the limpid solution obtained by filtration is contacted, without filtration, with malt ~~germs~~ sprouts

directly in the fermentation vat or, for the purpose of introducing nitrogenous elements, it is mixed with clear extracts or nutritive salts.

3. Another mode of carrying it into practice, in which the formation of the lactic acid is effected by the bacterial method, is characterized in that the clear and filtered molasses solution, with which clear nitrogenous extracts have been mixed and to which has been added a starter in the form of clear must has been added, is subjected to acidification for some time whereupon, as soon as the desired degree of acidity is reached, it is subjected to fermentation.

4. The very fine impurities which are held in suspension in the molasses solution are eliminated by filtration on large filtering surfaces in the absence of pressure.

9778.1

SWISS CONFEDERACY

FEDERAL OFFICE FOR INTELLECTUAL PROPERTY

PATENT SPECIFICATION

No. 65,539 CLASS 33 a

May 27, 1913 7 p. m.

PRINCIPAL PATENT

ALFRED POLLAK, THE ALFORT FIRMS,
IN THE OUTSKIRTS OF PARIS (FRANCE)

PROCESS OF ACIDIFYING FERMENTING SUBSTRATES
BY THE USE OF
AMMONIUM COMPOUNDS

In a plurality of processes of the fermenting industry a natural acidification is produced in the fermenting substrate, for example, in the mash in order to increase the fitness of the liquid, for example, for the subsequent alcoholic fermentation by means of

yeast. The acid formation, however, stops at a degree of acidity dependent upon the temperature and the kind of acid bacterium. If it be desired to push this formation of acid further and also to impart greater intensity to the physiological processes taking place at the same time, the already existing acid concentration which interferes with the fermentation, must be reduced by neutralizing by means of an alkali.

True enough, it is already known to add ammonium compounds, e. g., ammonium carbonate, to the fermenting substrates for increasing the nitrogen content of the fermenting substrates. As a result of the addi- soluble tion, ∧ salts of the acid formed by the fermentation are produced which not only promote the often necessary diastase effects in the processes under consideration, but also constitute excellent nitrogen-supplying nutrient salts for the growing yeast. It is however, new to use the ammonia compounds, e. g., ammonia or strongly alkaline ammonium compounds for instance, in a manner such that they are repeatedly added to the fermenting substrates with the object of neutralization repeated partial ~~saturation~~ of the acid resulting from the fermentation. It is an advantage of this process that far more acid is produced than in the former processes and that there results a correspondingly greater amount of soluble ammonium salt and pressable yeast, the former acting as an yeast nourishing and stimulating agent during the manufacture of alcohol for instance.

EXAMPLE 1:

To a mash prepared from any kind of raw material is mixed a pure culture of acid-forming bacteria, whereupon it is subjected to acidification at a suitably high temperature. When the acid formation has

reached a maximum, a test sample of the mash is titrated in the laboratory with alkali to the point of neutrality against a definitely selected indicator, with phenolphthalein for instance. The quantity consumed for neutralization is recalculated with respect to the entire mash, and to the latter is thereupon added so much ammonia that the amount thereof is sufficient for neutralizing ½-¾ of the amount of acid present. In this partly neutralized mash the acid-forming bacterium continues its work until the maximum of acid that can be attained thereby is attained once more. Thereafter the process of neutralization is repeated a number of times until even the increasing salt concentration causes the fermentation process to come to a stop. In this manner however, the formation of acid can be pushed to the quadruple of the former degree of acidity.

As neutralizing agents may be used ammonia, ammonium carbonate, salt of hartshorn, ammonium carbamate or hexamethylenetetramine. The latter of course will be prepared prior to its use most simply by mixing ammonia and a formaldehyde solution.

EXAMPLE 2.

A mash is acidified just as in the preceding example, except that prior to the neutralization of the acid by means of ammonia the mash is divided into two parts, one part of which is neutralized completely, and then admixed with the non-neutralized part which constitutes ½ to ¼ of the entire quantity. In this manner the operation is rendered safer, and impairment of the fermenting power of the acid-forming bacterium by over-neutralization is prevented. The mash thus prepared can then be used for the production of yeast or spirits.

Instead of the mash a solution of sugar or other fermentable substrate of low or high concentration can

be taken and the aforedescribed enrichment in acid be performed therein. Products obtained in this manner can advantageously be used as adjuvants for fermentations of any description, for example, as an auxiliary in baking.

PATENT CLAIM

Process of acidifying fermenting substrates by using ammonium compounds, characterized by this, that the latter are repeatedly added to the fermenting substrates for repeated partial neutralization of the acid resulting from the fermentation.

TRANSLATION OF FRITSCHE AUSTRIAN PATENT.

Austrian Privilege No. 45/4047
Innovations relating to the production of alcohol and pressed yeast.
Gustav Fritsche, Distillery Technician in Strzebowitz near Schönbrunn (Austrian Silesia)

granted on ..... Oct. 26, 1895
Priority of ..... Oct. 16, 1895
Expires ..... Oct. 6, 1900

The innovations forming the subject matter of the present invention relate to the use of ammonium salts, particularly ammonium sulphate, for the production of pressed yeast and alcohol, and have for their purpose to bring about a multiplication of the yeast cells, respectively a higher activity of the diastase.

Ammonium salts, particularly ammonium sulphate, serve as nutrients for the yeast cells and, when added to the pressed-yeast mash, thereby bring about a remarkable multiplication of the yeast cells. The utilization of ammonium sulphate promotes the activity of diastase in grain and potato mashes and effects an increase in yield in their case as well as in that of molasses mashes and sugar solutions.

According to the invention ammonium sulfate in an amount equal to a certain percentage (for example 0.5-1.0%) of the weight of the grain to be processed, is added to the cold water required for mashing corn or rye in pressed yeast manufacture. Depending on local conditions, the amount of ammonium sulphate to be added may even go up to 2% if the yield figures or the cost of production will allow to do so.

This mashing operation is carried out a few hours before the maize is boiled, so that at the same time a quicker and more complete solubilization of the latter is effected. Solubilization having thus been effected the mass of boiled corn is cooled down to 53-55°R. and mixed with the rye and malt, likewise steeped in water. This causes the temperature of the entire grain mixture to drop to about 40°R. and after proper commingling the mixture must once more be raised to the saccharification temperature of 50-51°R., this being effected by the admission of steam during which the mash must be maintained in a state of continuous rotary motion by the agitator.

The rest of the process remains the same as that in use for the production of pressed yeast. The result of the utilization of ammonium salts, particularly ammonium sulphate, is an increase in yield of the most durable and powerful pressed yeast. With the spent-wash process the yield is increased by 2½-3½%, with the ordinary process by 7 to 11 or 12%.

The ammonium salts, particularly ammonium sulphate, are used in the same way for the manufacture of artificial yeast. The amount of ammonium sulphate to be added is best calculated in terms of % of the capacity of the yeast fermentation vats. As a rule it is appropriate to take 0,4-0,6% of the latter. If, for example, the yeast-fermentation vat has a capacity of 300 liters, 1,2-1,8 kg. of ammonium sulphate would suffice in most cases.

A few hours before the so-called "pitching of the yeast" the ammonium sulphate is added to the mash or to the yeast product obtained by peptonization by means of hydrochloric acid. This addition of ammonium sulphate is an excellent nutrient for the yeast cells, which multiply considerably so that a much more intensely acting ferment is obtained which is capable of effecting a more complete conversion of the sugar into alcohol and which enables a higher yield to be obtained. The ferment produced in this way is equally well adapted for any kind of mash.

The ammonium sulphate promotes the activity of the diastase during the conversion of starch substances into sugar and is therefore utilizable with great advantage for the production of alcohol from starchy materials. For this purpose 0,25% to 0,50% of the amount of grain to be processed suffice to increase the yield of alcohol by 5%, frequently even by 8%.

The novelty of the subject matter of the privilege resides in the following:

1. In the production of pressed yeast or artificial yeast used for effecting alcoholic ferementation in the production of alcohol, the addition of ammonium salts, particularly ammonium sulphate, to the water used for mashing starchy substances for the purpose of obtaining an increased yield of yeast having good keeping qualities and a high rising power by means of these salts serving as nutrients for the yeast cells.

2. In the production of alcohol from starchy materials the addition of ammonium salts, particularly ammonium sulphate to the mash, for the purpose of intensifying the starch-saccharifying action of the diastase.

XVII.—BREWING, WINES, SPIRITS, ETC.

Yeast; Development of ——, in Sugar Solutions without Fermentation. Iwanowski, Centr. Bl. f. Bakter, u. Parasitenk, 10, (2), 151-154. Chem. (Centr., 1903, 1, (15), 890.)

The author has previously found that (1) the higher the concentration of nitrogenous substances (peptones) the weaker is the fermentation; (2) the higher the concentration of sugar in the nutrient solution, the stronger is the alcoholic fermentation; (3) in solutions which contain about 0.5 per cent of sugar and about double that quantity of peptone, hardly any alcoholic fermentation is to be observed. In view of Richter's statements (this Journal, 1902, 1239), the author has repeated the experiments which led him to the above conclusions, and he reiterates his statement that in solutions rich in nitrogenous substances, yeast loses in a high degree the character of a fermenting organism, whilst, on the other hand, its reproductive power becomes greater, notwithstanding access of air, and other experimental conditions remain the same. The influence of the concentration of sugar was also examined, and it was found that yeast can assimilate sugar without causing alcoholic ferementation. The free access of air is, however, not alone sufficient to bring about this state of affairs; it is also necessary to have a nutrient solution of suitable composition. The author considers that Pasteur's theory of the relations between alcoholic fermentation and oxygen must be extended in accordance with the above facts.

—A. S."

May 30, 1903, Journal and Patent Literature— Cl. XVI &XVII. p. 643.

XVII.—BREWING, WINES, SPIRITS, ETC.

Yeasts: Influence of meal and other nitrogenous materials, salts and acids on the life and fermenting power of ..... in distilled water with sucrose and in worts. W. Henneberg. Woch. f. Brau., 1908, 25, 77-81, 99-104-118-123, 138-141, 161-164.

The author has carried out an extensive series of experiments which show that rye or wheat-meal, egg albumin peptone, lecithin and inorganic ammonium salts exhibit striking similarities in their action on yeasts. These substances are: (1) non-poisonous to yeasts in pure, distilled water; (2) more or less poisonous in distilled water in presence of sucrose; (3) non-poisonous in supply water; (4) non-poisonous in sucrose solutions, if calcium salts, carbonates or bases are present in small amounts or other salts (such as acid potassium phosphate, magnesium sulphate, sodium sulphide, sodium formate or potassium sulphate) in higher concentrations; (5) of great nutritive value in presence of the substances mentioned under (4). Further, rye or wheat-meal, egg albumin, peptone, ammonium citrate and ammonium oxalate have a much more powerful poisonous action on bottom-fermenting beer yeasts than on top-fermenting distillery yeasts; this is also the case with lecithin, though in a less marked degree. The inorganic ammonium salts have an approximately equal poisonous action on both kinds of yeast. Under ordinary conditions, these nitrogenous substances form excellent nutrient materials for yeasts, their injurious action only appearing in the absence of certain salts, which are more especially compounds capable of neutralizing acids. The other salts able to annul this injurious action of the nitrogenous substances comprise all the calcium salts with the exception of the oxalate and, in much greater concentrations, magnesium sulphate, ammonium sulphate, potassium

phosphate, etc.; the experiments show that calcium and certain other salts are capable of acting to some extent, on acids in such a way as to render them less harmful to the yeast. It must, therefore, be assumed that the nitrogenous compounds give rise, in the interior of the cells, to free acids, which, under normal conditions, are immediately rendered innocuous by the above-mentioned salts; if such salts are lacking, the acids exert a poisonous action on the cells. In the case of ammonium salts, these must undergo decomposition in order to take part in the formation of proteins, the acid of the ammonium salt being liberated; if, therefore, this acid has a poisonous action on yeast, the corresponding ammonium salt will have a similar action. Similarly, lecithin owes its poisonous action to the phosphoric acid set free. Experiments on the length of time required by the different nitrogenous substances to act on yeast, show that the soluble proteins are the most rapidly assimilated and decomposed by the yeast, the action being slower for ammonium salts; the rapidity with which yeast dies under the action of rye or wheat-meal is possibly due to the large quantities of nuclein substances present. Distillery yeasts are considerably more resistant to the action of acids than are bottom fermenting beer yeasts. The remarkable fact that small quantities of ammonium salts exert a greater injurious effect than larger quantities is possibly due to the more rapid assimilation of the smaller amounts by the yeast cells; perhaps also the harmful action of distilled water is reduced when larger quantities of salts are present. —T. H. P.

of the mash rises to 31° and may even go up to 34° or 35°C. Spirit distillers should pitch as low as possible and take care that the temperature rise during fermentation does not pass beyond 30°C. As regards the quantity of yeast to use the rule applies: If much seed yeast is used the yeast crop will be smaller, but the fermentation will be the sooner ended.

Souring With Mineral Acids.

The lactic acid process can be dispensed with in favor of the direct addition of sulphuric, phosphoric, oxalic, tartaric, technical lactic acid, hydrofluoric acid or its salts, or sulphurous acid. In making use of these acids the souring room and its inconveniences become unnecessary, that is, the operation of the factory becomes simplified. In general all the well known methods of preparing seed yeast are about equal in value, provided they are applied and carried out with the necessary attention. In passing from the one method to the other, the distiller may notice that his yields, when he is working with a given kind of raw material, may either be increased, or may remain the same, or may fall off. The question as to which process should be adopted should be decided in view of the equipment of the distillery and general convenience in operation.

Instead of a mash prepared from malt or other grains, use is often made of malt sprouts, or living or dried beer yeast, as yeast food, with or without addition of other mineral yeast foods like phosphoric acid or its salts. For purposes of sterilization the malt sprouts are either heated with the yeast mash, or, what is better, they are put to soak the day before use with sulphuric acid. For every 25 gallons of yeast mash we take two pounds of malt sprouts, and the mash is acidified by the addition of 80 or 130 cubic centimeters of concentrated sulphuric acid which has been previously diluted with six or seven times its volume of water. The malt sprouts may be allowed to soak in this mixture for from 24 to 72 hours; during this time the bacteria adhering to the malt sprouts will be killed and the sour liquid will dissolve as much albuminous substance as could be obtained from dried beer yeast.

One of the chief errors of practical men is to anticipate the effect of an unusual increase in the acidity of a sour mash by the addition of more acid without taking the trouble to ascertain the cause of the increase. Often the yeast is seriously weakened by the addition of too much acid; this means too much acid in the main mash also, and the result is a diminished yield of alcohol. Where this occurs the yeast must be replaced by fresh pitching yeast. To bring a weakened yeast again into a vigorous condition is a more expensive and time-consuming operation than to procure a new mother yeast. If, however, the yeast is a specially good one, and has not become too much weakened or impure, it is worth while to refresh it.

The normal amounts of acid specified in the newer processes of Buechler and others assure a medium or average yield, but the distiller or yeast manufacturer should from time to time make a special test to ascertain the most favorable degree of acidity for the raw materials he is using. Every 0.1 degree of acidity above the optimum has an unfavorable effect on the alcohol yield. The fermentation in the main mash is perceptibly weakened and delayed when there is as much as 0.3 or 0.4 degrees of sulphuric acid. In making a sour yeast mash with sulphuric acid, no more than 1.3 degrees of this acid should be used; above this limit the yeast suffers a decrease of activity. When the activity is 1.8 or 2.0 degrees, the weaker yeast cells die off and even the stronger cells live only 24 or 48 hours unless immediately brought into the prepared sweet main mash, where they multiply and again begin a vigorous fermentation. Two or three degrees of acid are eventually fatal to the yeast.

Yeast of itself produces considerably less acid in acid media than in neutral media, and in this respect resembles the acid producing bacteria. Unless this point is regarded mistakes may easily be made.

One may work according to the Wilfert or other sulphuric acid methods which specify from 0.8 to 3.6 degrees of acidity, but these methods are uncertain and full of trouble.

If one is working with a pure yeast, the aim should be to work with as little acid as possible, and resort to purifying the yeast with sulphuric acid only occasionally. If the yeast is not free from bacteria the distiller may begin with 1.0 or 1.3 degrees of acidity and gradually decrease it. One may also proceed by adding 2.0 to 2.5 degrees of acidity to the pitching yeast and allow to stand for half an hour.

There are some yeast makers who add as much as 6.0 degrees of acid to their yeast mashes, but in such cases they must add the yeast mash to the main mash within 10 minutes or the yeast will perish. Some yeast makers use hydrochloric acid to the extent of 2.2 degrees, allowing two hours as the minimum time for the acid to act.

The Distillation of Final Molasses

By PROFESSOR WM. L. OWEN.

Bacteriologist Sugar Experiment Station, Louisiana State University.

CHAPTER V.

Sluggish Fermentations of Molasses; Their Causes and Means for Their Prevention.

When operating a distillery under the seventy-two hour law, which applies to all such industries throughout this country, any tendency toward sluggishness of fermentation can only result in a de-

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Vol. IV. **MAY 1908** **No. 5.**

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mentation. As wines are usually poor in nitrogen, there is added as nutrient for the yeast about 20 gr. of ammonium chloride, which is completely consumed by the yeast.

Thus it is apparent that by the employment of pure culture as contrasted with the old method of spontaneous fermentation, very significant advantages may be secured, of which the most important undoubtedly is, that the wine producer has, so to speak, the fermentation in his hand, and is not dependent upon the hap-hazard results of accident and chance. There is, accordingly, not the slightest doubt that the utilization of pure yeast, as has been introduced into the brewery long ago, is also in the production of wine destined to bring about a complete revolution.

DR. W. DONSELT.

On the Influence of Flour and other Nitrogenous Substances, Salts, and Acids on the Duration of Life and Fermentative Power of Yeasts in Distilled Water with Saccharose, and in Worts.

UNDER this title, W. Henneberg has published his reports of investigations made in the technico-scientific laboratory of the Institute for Fermentation Industries in Berlin. In No. 10 of the "Wochenschrift fuer Brauerei", current volume, he summarizes the results of his exhaustive investigations in the following conclusions, which are unquestionably of great interest:

CONCLUSIONS.

From the reported results of investigations it is apparent, that flour, egg albumen, pepton, lecithin and inorganic ammoniacal salts in their influence upon yeast, exhibit striking resemblances, which above all, show themselves in the following particulars:

1. Not "poisonous" or "toxic" in pure distilled water.
2. More or less "toxic" in distilled water with sugar infusion.
3. Not toxic in hydrant water (of the Institute) with sugar.
4. Not toxic, as soon as lime salts, carbonates or bases are present in the sugar solution in very slight amount, or other salts in greater concentration.
5. In the presence of these substances they are mostly of excellent nutritive value.

6. Flour, egg albumen, pepton, citrate and oxalate of ammonia, kill off the under-fermenting beer yeast in much more vigorous degree than they do the top-fermenting distillery-yeast; with lecithin this is likewise the case, though the phenomenon here is not so clearly apparent. Only the case, though the phenomenon here is not so clearly apparent. Only the inorganic ammoniacal salts are poisonous for both yeasts in approximately the same degree.

From the chemico-physiological point of view the above mentioned substances have this in common, that they are all nitrogen compounds, which have long been known as excellent yeast nutrients under ordinary conditions. The assumption is, therefore, probably valid, that they all exercise an injurious effect in the same manner and for the same reason, as soon as certain salts are lacking.

If, in order more exactly to establish the cause of their toxicity or injuriousness, we examine the kind of substance necessary to render them innocuous, we notice, that they are, above all, acid-destroying substances, viz., bases and carbonates.

We may, however, further classify among these all the lime salts—with the exception of the oxalate of lime—and so far as observations have gone, in much greater concentration, magnesium sulphate, ammonium sulphate, potassium phosphate, etc. Do these also operate upon acids within certain limits, so that they are not at all, or only slightly injurious in effect? So far as experiments have up to this time been carried out, this has been definitely established in the case of lime salts, and likewise for several other salts.

We must therefore assume, that in the process of conversion of the nitrogen compounds named, free acids are generated in the interior of the cell, which are normally rendered innocuous imediately by the salts just mentioned. If the salts are lacking, the acids may be deadly in their effect (c. f. address of the author before the October Meeting, 1907, 1907 Volume, Page 638—650).

The conditions in the case of ammonium salts are very simple. The yeast, as is well known, may with great advantage use them for its production of albumen. For this purpose, it must of course split them up. The acid which has been in combination with the ammonia is thus liberated. If the latter is carbonic acid, no poison develops. If they are only slightly injurious organic acids, they may be rendered innocuous by combustion. Citric and oxalic ammonia salts have a poisonous effect on beer yeast, because these acids are extremely toxic for this yeast. For the same reason, the inorganic ammoniacal salts, with the exception of the carbonate of ammonium, are extremely poisonous in effect.

A similar explanation might be given of the toxicity of lecithin. The yeast in this case probably dies off because of the phosphoric acid being liberated.

In the splitting up of pepton, ovalbumen and flour, besides various organic acids, oxalic acid, sulphuric acid and phosphoric acid might arise. As lime salts not only render oxalic acid innocuous, but exercise this same effect on sulphuric acid, phosphoric acid and other acids, there is no way of determining in each individual case, which of these acids is in question before us. Against its being oxalic acid, there speaks to a certain extent the fact, that in spite of many experiments where a subsequent lime infusion, etc., was made, only very rarely could oxalate crystals be observed in the interior of the yeast cell. Probably in some cases (flour) phosphoric acid developes besides organic acids, and in others (pepton), sulphuric acid. It probably cannot be determined by chemical analysis, for naturally only very minute quantities come into question, which possibly do not force their way out of the cell at all.

If we observe the period of time during which the substances exert their influence, it becomes particularly striking, that the flour operates injuriously most rapidly of all, pepton and ovalbumen somewhat less quickly, and the inorganic ammoniacal salts only after several hours. In this very order, we probably have before us the order of succession of the utility (assimilation) of these nitrogen compounds for the yeast; the soluble albumen compounds are taken up by the yeast and decomposed with the greatest rapidity, and the ammonium compounds more slowly. Possibly the prolific quantities of the nuclein materials of the flour are also at the same time the cause of the rapid dying off of the yeast.

A difference between distillery yeast and bottom-fermenting beer yeast is in most cases clearly apparent under the influence exerted by flour, ovalbumen, and pepton, in that only the bottom-fermenting beer yeast is destroyed. Inorganic ammonium salts kill both, though to be sure after some hours only. Citrate of ammonium destroys beer yeast more than it does distillery yeast. Oxalate of ammonium only destroys beer yeast! Through many comparative experiments, the fact was definitively proved, that distillery yeast possesses a considerably greater power of resistance against acids (sulphuric acid, phosphoric acid, etc.) than does the bottom-fermenting beer yeast. Against some acids such as acetic acid, butyric acid, valerian acid, and oxalic acid, distillery yeast is conspicuously more capable of resistance, and against tartaric acid and citric acid, has much greater power of resistance, than beer yeast. If it is certain, that in the albumen decomposition in the organism, acetic acid, butyric acid, valerianic acid, oxalic acid, etc. are also generated, that is to say, acids

to which the bottom-fermenting beer yeast is very susceptible, and against which the distillery yeast offers great resistance, the difference in their behavior toward flour, pepton and egg albumen would be easily explainable. From the fact that the inorganic ammonium salts are equally poisonous for both yeasts, we could also conclude with some probability, that the question is solely, or at least very prominently, one of organic acids in the decomposition of albumen, which accounts for the differences in the behavior of beer yeast and distillery yeast.

Flour, ovalbumen and pepton are deadly only in the beginning upon beer yeast; indeed later on, they exercise a stimulating influence upon the surviving cells. Ammoniacal salts on the other hand, only begin to have an injurious effect after some time of operation, but then, however, it is permanent. We can only explain this by assuming, that the ash content of the first mentioned substances, by decomposition for example, gradually gets into solution or into some other suitable form in which it may then be taken up by the yeast cell. A great deal of pepton is therefore, not as "poisonous" as only a little pepton. From the dead cells materials are also gradually excreted, which are consumed by the surviving cells.

The very remarkable observation, that small quantities of ammoniacal salts have a more injurious effect that definite larger quantities may possibly be explained by the fact, that the yeast cell takes up the former more easily than it does the latter. Moreover through greater quantities the injurious effect of the distilled water itself is possibly more fully restricted. In this latter way we may probably also be able to explain the fact, that many salts in considerable concentration, may pretty strongly diminish the effect of the operation of flour.

As was shown in the author's earlier investigations on nutrient solutions (Wochenschrift fuer Brauerei, No. 40 to 45), acid ammonium salt solutions with the other necessary nutrient salts are extremely unfavorable for bottom-fermenting beer yeasts and moderately unfavorable for distillery yeasts. As soon, however, as chalk, soda, or lactate or phosphate of lime is added, these solutions in all cases become very favorable, whereas gypsum and calcium chloride infusions exercise only a moderately good influence upon bottom- and top-fermenting beer yeasts, and upon distillery yeasts no noticeable influence at all. Inorganic ammoniacal salts without lime salts or bases are, therefore, injurious.

Pepton always operates favorably in nutrient solutions, and likewise when it is added to the wort. Possibly only a slight amount of acid is generated from the pepton. It would thus be explainable, that the injurious effect of pepton in sacchariferous distilled water very soon disappears again.

Flour operates very unfavorably upon bottom-fermenting beer yeast in worts that are concentrated and diluted with distilled water. The necessary lime salts are also here lacking, as the effect disappears immediately upon the addition of lime salts. Gradually, however, the surviving cells begin to sprout very prolifically and very vigorously to ferment. We may, perhaps, here also assume, that the lime salts from the flour, are not at first, but only later after solution and conversion, at the disposal of the yeast cells and that from the many dead cells salts are also lixiviated, which are taken up by the living cells.

How now shall we explain the phenomenon, that chalk, soda and in fact basic substances, so very strikingly delay the death of yeast cells in worts (without further infusions)? The acid of the wort was considered heretofore as obstructive of development and injurious for innumerable varieties of bacteria only. It unquestionably also so operates, even though in much slighter degree, upon yeast. The acids, which develop as products of assimilation of the yeast, will likewise exercise an unfavorable influence.

The fact that the flocculation of the beer yeast and, connected therewith, the clarification of the wort, is better and makes its appearance more quickly if the substances named are added to the wort, is accounted for in the first instance by the fact, that the acid of the wort has become diminished. Lime salts (also organic) have quite commonly a favorable effect also upon the slime development of the beer yeast; perhaps this is also the case with sodium salts.

The yeast cells in saccharose solutions in distilled water, die off to the extent of half and more, after 24 hours. All the materials which suspend the effectiveness of the flour, etc., here also prevent the annihilation. We undoubtedly have before us here the same phenomenon, only that the yeast decomposes its own voluminously stored up albumen, and from this permits acids to generate. At this occasion, fusel oil, as Ehrlich assumes, is also generated through the decomposition of the amino-acids.

Nutrient solutions with ammonium-nitrogen are apparently always characterized by their fusel oil content, (so far as we may conclude from their odor). Experiments have still to determine, whether fusel oil is actually present in these cases in prolific amount. Possibly we have here another important contribution to the explanation of the formation of fusel oil.

It may be remarked in this immediate connection, that the yeast, as we could observe, was very easily able in characteristic manner, to decompose inorganic salts also. As is well known, sulphurretted hydrogen developes from magnesium sulphate in considerable quantities, in

nutrient solutions. We moreover observed the formation of sulphurretted hydrogen from potassium sulphate, calcium sulphate, ammonium sulphate, calcium sulphite, as well as from gypsum in the presence of sulphuric acid.

From both ferous prussiates of potash and nitro-prussiate of soda, yeast is able to liberate hydrocyanic acid.

Berlin White Beer.

BERLIN white-beer (Weissbier), a top-fermented beer, has been known for centuries, and is brewed in Berlin as a specially characteristic variety of beer, out of wheat- and barley-malt. Since the sixties of the last century, a beer has been brewed very similar to that produced to-day, whereas in the production of the old white-beer a smoked wheat-malt was employed, this custom being due to the fact that at that time, the smoky taste of the beer was extremely popular.

Berlin white-beer, as we have said, is a top-fermented beer, whereas Munich, etc., are bottom-fermented beers. After the fermentation, two different courses may be pursued in the method of procedure for the preparation of the beer. In the first part of the operation, namely until the middle of the fermentation period, a common course is followed. Whereas in the bottom-fermentation process, the yeast in the beer gradually settles at the bottom in a solid layer, being then removed from the vat after completed fermentation, subsequent to racking of the beer into the storage tanks, the procedure in the other manner of fermentation, the top-fermentation, is so carried on, that the yeast cells, engaged in the most vigorous fermentative activity, are drawn toward the top, and at the surface of the beer, form a tough, white, and quite consistent yeast-layer.

About one-fifth of all the beers brewed in Germany, are brewed under top-fermentation; these are Graetzer beer, Gose, Lichtenheimer, Berlin Weissbier and many others. In England this group is increased by the characteristic English beers, as porter and ale. All our lager-beers, on the other hand, are bottom-fermented. The yeast race is the determining factor of a top-or bottom-fermented beer, depending upon whether a top-fermenting or bottom-fermenting yeast is added.

Berlin white-beer is a peculiar and quite characteristic variety of top-fermented beers. In its production, wheat- and barley-malt are

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TRANSLATION OF PAGE 562

From: Centralblatt f. Bakt. etc. 1914 an article entitled "The Nitrogen Nutrition of Mold Fungi", starting on Page 555.

As shown by Czapek (I), salts of hydroxylamine and hydrazine can serve as N-sources, although only in case of necessity.

For the purpose of clarifying the question as to whether amino acids and amides can be directly assimilated or whether their decomposition into acids and NH_3 must first take place, and also in order to learn the connection between the working of these compounds and the production of oxalic acid, the writer has prepared numerous cultures. In most of these solutions he found NH_3 even in the presence of an abundance of sugar as the source of C and from this he concludes that deamidization precedes assimilation. He could find no connection between the N-assimilation from amino acids and the formation of oxalic acid. As regards the importance of amino-acids for the synthesis of the proteins Raciborski accordingly takes a different standpoint from that of Czapek inasmuch as he denies direct applicability to them in the synthesis.

As was to be seen from the foregoing, aspergillus niger or some perisporiceae closely related to it was used as the object of the investigation. The chapter in O. Hagems "Researches on Norwegian Mucorineae" which deals with the N-resorption of these fungi, is therefore of great interest. His careful, extensive investigations deal mostly with N-compounds, of which it can be assumed that they play a role in the life of the mucorineae in nature.

Nitrite and nitrate can be utilized by a limited number of species. Owing to their capability of reduction to NH_3, HNO_2 and NH_3 can mostly be detected as reduction products of nitrates, NH_3 as the reduction

products of nitrites. The reduction to NH_3 is to be regarded as an intracellular phenomenon.

Inorganic NH_4 salts are good sources of N (nitrogen), provided the accumulation of free mineral acid is prevented by the addition of $caCO_3$. NH_4NO_3 occupies an exceptional position with respect to those species which reduce nitrate. Both the NH_4 and the NO_3 ions are utilized by them about equally quickly.

To the extent as organic NH_4 salts have been tested, they constituted an excellent N-nutriment. Urea is worked up by all the species investigated. They produce varying, and often quite considerable, quantities of $(NH_4)_2CO_3$. Acetamid has an insignificant value as a nutriment or none at all. Uric acid is used with success. The separation of NH_3 could not be ascertained with certainty, but it probably takes place nevertheless. Amino-acids are very good sources of N in the presence of sugar. In all culture liquids NH_3 was detected in varying amounts. The same applies to peptone, which besides can be used with advantage as a source of C (carbon).

Hippuric acid is worked up by a relatively small number of species. It splits up into benzoic acid and glycocoll, and the latter compound is further broken down with formation of large amounts of NH_3.

As can be seen, the separation of NH_3 accompanies the assimilation of N almost without exception. From this the writer draws the conclusion, which has been already pointed out by Raciborski and other writers with regard to the working up of amino-acids by aspergillus, that "the fungi must form ammonia in any nitrogen resorption and are able to begin their protein synthesis only from the former as a starting point".

9778.

A. WILFERT

PRESSHEFE, KUNSTHEFE UND BACKPULVER
VIENNA, 1890, 2nd Ed. pp. 192-195 incl.

The (proper) quantity of water is placed in a special vessel (water at ordinary temperature is used) and the weighed amount of sulphuric acid is poured in a thin jet into the water which is vigorously stirred with a wooden stick during the pouring. In consequence of the chemical combination into which the sulphuric acid enters with the water, the whole liquid is heated so strongly by the liberated heat that it begins to give off steam.

Let it be expressly stated here that the sulphuric acid must always be poured into the water; if, conversely, the water were poured into the acid, this might have the most dangerous consequences as so much heat would then be set free that part of the liquid would be set into violent ebullition and hot acid be ejected from the vessel. Persons struck by this acid suffer serious injuries. If one works in the way indicated, that is, if the acid is poured into water set in motion, the operation of the two substances is attended with no danger at all.

The mashing of the malt and grain grist with water at 48 to 49° R. is continued until by means of the iodine solution test one has become quite sure that no unchanged starch remains in the liquid. The iodine test consists in adding a few drops of a solution of one part of iodine to 20 parts of alcohol to a small quantity of the mash clarified by filtration and brought to room temperature. As long as the action of the diastase of the malt is incomplete, the liquor will keep on exhibiting a wine-red color; on the other hand, when on addition of the iodine solution the liquid remains colorless, then the mashing operation is to be regarded as finished.

Only then is it permissible to add the sulphuric acid, as the saccharifying action of the diastase on the starch would be disturbed by the premature addition of the acid. The relative proportions of finished mash and sulphuric acid depend on the amount of the material (malt and grain together) used for the preparation of the mash. To every 100 kilograms of material one takes one kilogram of sulphuric acid which is diluted with 5 litres of water, as stated above.

The diluted sulphuric acid is slowly run into the hot mash and most intimately mixed therewith by constant stirring, whereupon the temperature of the entire liquor is allowed to drop to 36° R. and maintained there for three to four hours.

There now takes place in the mash exactly the same reaction which takes place when a mash is subjected to a lactic acid fermentation; the proteins present in the malt and raw grain dissolve and are converted into peptones, and after four hours the starting yeast can be considered to have reached full maturity and the mother yeast is added thereto.

The mash, however, contains a rather large amount of sulphuric acid, and after cooling the mash to 18 to 16° R. this acid would have a disadvantageous influence on the mother yeast which is now to be added; it is therefore necessary to bring the sulphuric acid content of the liquor to a lower figure without it being necessary, however, to separate the sulphuric acid from the liquid.

The sulphuric acid acts disadvantageously on the development of the yeast only when it is present in large amounts; conversely, a small amount of sulphuric acid is highly beneficial for the development of the yeast, as it contributes considerably to the production of a very pure and vigorous yeast. The yeast plant requires for its vigorous growth a liquid which is slightly acid; apart from the fact that this constitution

of the liquid promotes the development of the yeast plant, it renders the development of fission fungi almost entirely impossible, inasmuch as the latter, as experience has shown, can thrive well in neutral and alkaline liquids but cannot develop in acid liquids.

If, therefore, for the mash that moment has arrived where the action of the sulphuric acid on the proteins can be regarded as completed, then according to the usual processes the cooling to 18 to 16° and the addition of the mother yeast would have to be effected; however, it is better to effect no special cooling but to proceed in the following way:

The preparation of the starter treated with sulphuric acid is effected in a vessel capable of holding three to four times as much liquid as the amount of starting yeast to be prepared at one time, and into the finished mash, which still has a temperature of 36°, is run so much sweet mash held at ordinary temperature that the temperature of the mixture drops to 16°, after which the mother yeast is added.

In a mash in which the sulphuric acid content is diluted to that extent, the development of the yeast and the fermentation proceed in a perfectly normal way, and there is no reason to be found for removing the sulphuric acid from the liquor. This can be done in a very simple way by adding to the mash slaked lime—stirred up in water to give a milky fluid—until the acid taste of the mash is scarcely discernible any longer.

The lime combines with the sulphuric acid to form calcium sulphate or gypsum, which compound is only slightly soluble in water and accordingly separates from the liquid in the form of a white powder. After the addition of the lime the liquid must be left undisturbed until the lime has settled out at the bottom, after which the liquor standing above the sediment is carefully drawn off.

As one can convince himself by a comparative test with two lots of the same mash, one of which was neutralized with lime whereas the other was left in the original state, the neutralization has not the slightest influence on the amount of newly-produced yeast nor its fermenting power and for these reasons the neutralization can be omitted entirely.

After the addition of the mother yeast to the sulphuric acid mash, the latter is further processed in the same way as with any other starting yeast; it is allowed to ferment to a definite degree, the newly-produced yeast is . . . .

9778. 93524C

TRANSLATION

Wochenschrift für Brauerei
1903, Jan. 18—20th year—No. 3; p. 25

SOME PRINCIPLES OF YEAST REPRODUCTION

By

M. Delbrück

1. Under similar conditions, particularly with mashes and worts of similar composition, the number of yeast cells in a given volume at the end of the reproductive period is the same.

2. Accordingly, the number of yeast cells sown does not affect the ultimate yield. With a greater number sown, a smaller ~~reproduction~~ multiplication occurs, with a smaller number, sown, a greater ~~reproduction~~ multiplication.

3. The cause of this phenomenon is to be sought in that the yeast cells mutually exert a disturbing influence. Each active yeast cell controls a certain space around it, in which it does not allow a new yeast cell to develop. The specific reason for this phenom-

enon is to be sought in that the excretion products of the yeast are injurious to the yeast itself, and, for that reason, to its neighbors of the same species. There comes into consideration mainly alcohol and carbonic acid, but also fusel oils, protein conversion products, etc. Since most of the carbon dioxide escapes from the liquor, the alcohol remaining in the latter is, for the main part, the controlling factor. In cold fermentations, such as in bottom-fermentation brewing practice, the alcohol content, however, is so small and the carbon dioxide content so high that in the present case the converse happens, e. g., the main role is played by the carbon dioxide. Carbon dioxide is ten times as poisonous to yeast as alcohol.

4. The individual yeast races present different degrees of susceptibility towards alcohol and carbon dioxide; this is the reason why one race stops ~~propagation~~ multiplication earlier, another one later. Of course, other excretion products can also be decisive.

5. The first principle set forth is only valid when the yeast cells or the liquid with the yeast cells is in uniform quiet movement, so that the cells are distributed uniformly throughout the liquid. If a part of the yeast is separated at the top by flotation or a part thereof goes to bottom by sedimentation, then multiplication of the cells remaining distributed in the liquid will be favorably influenced, as space is then available for them. This action will however, occur only until the maximum proportion of alcohol and carbon dioxide content permissible for ~~propagation~~ multiplication has been reached. For they are uniformly distributed in the liquid, their action is not influenced by the ~~separation~~ excretion

of yeast cells. Experiments are not yet available as to whether a larger yeast ~~propagation~~ multiplication can be obtained by the systematic separation of part of the cells already formed.

6. The main experiment, by which the basic law can be proved, consists in that a given volume of liquid is inoculated with different amounts of seed yeast. Microscopic observation shows that with a given slight seeding all the cells bud, and according to the strength of the seed, form one, two or more new cells, that furthermore, with increasing quantities of seed, the budding activity of individual cells is stifled (the strongest individuals ~~propagate~~ multiply) that, finally, with a very large amount of seed (10% of the weight of the liquid in pressed yeast) all the cells fail to bud. This experiment can be easily carried out in the laboratory, it is in agreement however, also with the observations made in practice, both in the worts of breweries and in the acidified culture liquids of distilleries and their main mashes.

7. If dissimilar nutrient liquids are present, then the following changes occur: In very dilute media, the number of cells produced in a given volume is less, owing to deficiency of nutrition. The number of cells increases with the proportion of available nutriment, but only as long as multiplication is not prevented through the accumulation of alcohol. As a rule, 4% of alcohol by weight suffices to put a limit to multiplication. Worts and mashes of a concentration which permits of exceeding this alcohol content, do not give the yeast multiplication corresponding to their nutrient content. To obtain the highest number of cells this alcohol content must not even approximately be formed during the fermentation. Manufacturers of yeast who

operate with worts, have a wort concentration of perhaps 7% on the saccharometer. With increased admission of air, as by using very shallow fermentation vats or by directly injecting air, the multiplication becomes greater. The stimulation of the budding activity by the oxygen of the air is so great that the higher number of cells is already attained before the inhibition due to the accumulation of alcohol sets in.

8. But even motion, particularly the motion in the earlier stages of the fermentation, leads to changes in the number of cells formed. Not only do the excretion substances act upon neighboring cells, but also upon the seed cells themselves. If motion is imparted to the medium, the individual cell itself is carried away from its station; it emerges from the excretion substance present in its immediate vicinity, it strips it off so to speak and reaches places yielding new nourishment which through the ready nutrients stimulate budding.

10. This initial motion can be brought about by an agitator; it can however, also be effected by the number of seed yeast cells and by the mode of this addition. Too small a quantity of seed yeast is the cause of a lessened multiplication, owing to a deficiency of carbon dioxide gas to induce the initial motion. If the addition is effected in such a way that the seed yeast is—to use the technical expression—applied in the form of a preliminary mash, i. e. instead of distributing pressed yeast in the liquid there is added an already fermenting medium pitched with seed yeast, motion will be present right from the start and that for the following reasons.

The given amount of seed yeast, to which a small amount of fermentable liquid has been added, induces at once an active fermentation in the latter and sets it

into motion since cells are present in excess. After the addition of the main liquid this strong motion will be transmitted to the latter, for the reason that all of the cells have already started budding, therefore a stronger fermenting action is present, and, furthermore, because not only have yeast cells been added but also a carbon dioxide-containing liquid. The highest crop therefore is obtained by the continuous process of fermentation according to which there is first placed into a large fermentation vat the inoculating yeast together with a little wort, whereupon a stream of fresh nutrient liquor is run in continuously, distributed over, say 24 hours, until the receptacle is full.

11. The nature and quantity of the excretion products depend also upon the physiological condition of the yeast cells, the ~~reproduction~~ multiplication, therefore, is also dependent on the physiological condition of the seed yeast.

Translation of Franz Lafar, "Handbuch der Technischen Mykologie", Fourth volume, 1905-1907; from page 131, line 10, to page 132, line 33, inclusive.

To hinder or stop fermentative activity, it is necessary to have, under otherwise equivalent conditions, a larger addition of alcohol than that which is able to stop the growth of the yeast cells. Thus a pressed yeast investigated by Hayduck (2) no longer increased in a 7 percent by volume sucrose nutrient solution, although lively fermentation continued. To hinder this also, according to the conclusions of an investigation by Brefeld (1), an alcohol addition of 17.3 percent by volume to the nutrient solution is necessary. On the contrary, in Hayduck's (3) investigation, absence of

fermentation was observed with only 15 percent of alcohol, by volume, in the nutrient solution. V. Peglion (1) found in an Italian wine during its after-fermentation an active yeast which still showed fermentative activity in the presence of 14.3 percent by volume of alcohol in the nutritive substratum. Further facts on the influence of the presence of alcohol on alcoholic fermentation will be found in Chapter 17. With reference to Mycoderma the 14th chapter may be consulted.

Of the other conditions which have a determining influence on the magnitude of alcohol concentration or addition which restricts fermentation, temperature is to be placed in first rank. Inside those limits which can come at all into consideration here, the sensitivity increases with temperature. With it there first increases, from the physical viewpoint, the permeability of the cell wall by means of osmosis for the alcohol tending to enter the cell's interior and, second, from the physiological viewpoint, the lability (ease of change) of the molecular groups of the plasma, as well as the sensitivity of these to outside influences. We owe to H. Müller-Thurgau (1) some facts about this. He found, under the conditions chosen by him for the experiments (that is, the character of the nutrient solution and the kind of strain of yeast) that under like conditions fermentation came to a standstill:

at 36° C. as soon as there was present 3.8 percent by weight of alcohol,
at 27° C. as soon as there was present 7.5 percent by weight of alcohol,
at 18° C. as soon as there was present 8.8 percent by weight of alcohol,
at 9° C. as soon as there was present 9.5 percent by weight of alcohol,

The existence of a relation between the heighth of the temperature and the degree of sensitivity of the yeast to alcohol seems to have been sensed by the old-time practical brewers, since they set down as the fundamental of their fermentation—not only for the making of good beer but for obtaining a strongly-fermenting setting yeast—the rule: Let the temperature in the vat rise at the beginning of the fermentation (when only a little alcohol is present) and then slowly bring it down.

The character of the nutrient solution equally exerts influence on the degree of sensitivity of the yeast to alcohol, therefore (from the viewpoint of fermentation technology), on the highest alcohol content attained by the fermenting liquid. Unfortunately, there are available in this field only a few errorless researches; like, for example, that of Müller-Thurgau (1). These have disclosed that under otherwise equal conditions, the injury caused by alcohol to the cell activity of the yeast increases with increasing sugar content of the nutrient solution. To this circumstance (not really understood in its cause but well known in its effects) there goes back the fundamentals of the process which under the name of Drauflassen ("addition") long has been used in various fermentation industries, especially in molasses distilleries, and has shown itself profitable. In those countries in which the brandy tax is levied entirely or partly on the size of the mash vats or fermentation vats, it is on this ground, to speak of nothing else, in the distiller's interest to mash his vats as thick (that is, sugar-rich) as is practicable. Thus, however, he comes in conflict with the effect of sugar concentration, already mentioned, in increasing the sensitivity of the yeast to alcohol. Here also the ingenious practical man, by many trials, has discerned a way out; he arranges such a

dilution of the molasses to be fermented as is known by experience to let the fermentation go on readily, and then adds thick molasses gradually, in the amount of the sugar that has disappeared. In those other distilleries which do not work on molasses but on unmalted grain (potatoes, Indian corn and the like) this Brauflassen is not very practicable, for reasons not to be discussed here. In brewing, on the other hand, it is not seldom resorted to, especially when the available amount of pitching yeast is insufficient and is to be increased quickly. (See the 6th chapter of the 5th volume.) In the sugar-rich musts used for the preparation of choice wines, which are fermented without the use of the expedient described, the fermentation remains stationary, as a result of the interrelations spoken of, before all the sugar is worked over, and one thus gets wines which actually have ceased to ferment but yet are sweet.

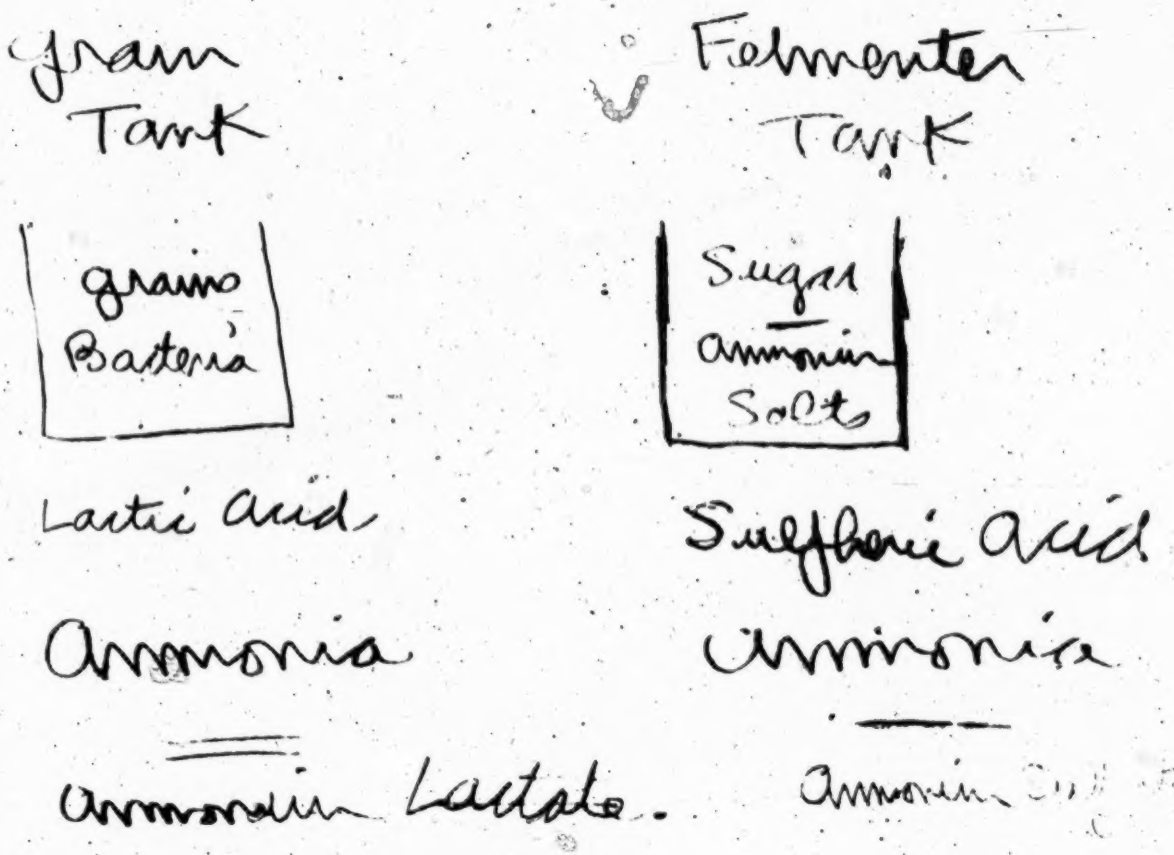
DEFENDANT'S EXHIBIT D-2.

Book Entitled Foreign Patents and Publications.

(Physical Exhibit.)

DEFENDANT'S EXHIBIT D-3.

Sketch of Grain Tank and Fermenter Tank.



Sketch of Grain Tank and Fermenter tank.

DEFENDANT'S EXHIBIT D-4.

Book Containing Records Concerning Experiments 3 and 4 Made May 31, 1935.

(Physical Exhibit.)

DEFENDANT'S EXHIBIT D-5.

Certified Copy of German Application Filed March 15, 1915.

(Translation Only Printed.)

SWORN TRANSLATION FROM GERMAN:

CERTIFICATION.

The photostatic copy attached hereto is a true and correct copy of the specification with claims, which has been filed on March 15th, 1915.

Berlin, this 10th day of March, A. D. 1935.

THE PRESIDENT OF THE REICHSPATENTAMT
(The Commissioner of the Patent Office of the German Reich).
As Substitute:
sgd.
Ellmenreich

L. S.
(RAISED SEAL OF THE PATENT OFFICE OF THE GERMAN REICH).

Pat. 303 251 (V 13 012 IV/6 a)

PROCESS OF MANUFACTURING PRESSED YEAST.

In the case of fermentations in solutions of sugar and salts, which are carried out in accordance with the process under the principal patent, a considerable increase of the acidity takes place in the fermentation liquid during the development of the yeast, without, however, the appearance of any bacteria. The acid thus created,—as has already previously been shown by Professor Dr. W. Henneberg,—is a mineral acid which apparently is produced due to the fact that the basic contents of the salts dissolved are absorbed by the yeast, and that the acid contents of said mineral salts are consequently set free.

Said free mineral acids exercise, as can also be observed by means of a microscopic inspection, a detrimental effect upon the yeast.

By the addition of suitable basic substances which are harmless, as far as the development of the yeast is concerned, as for instance carbonates, this mineral acid can be perfectly neutralised, or the acidity can, at least, be reduced to such a degree that it does no longer harm the yeast cells, whereby the detrimental effects occurring in this process are entirely prevented. Basic substances which are nitrogenous, can also be used. The process can, moreover, be made use of for the fermentation of pure sugar solutions with an admixture of mineral salts, without, however, a yield of yeast thereby being obtained.

Claims:

(1) In the process under the principal patent the neutralisation of the acid mineral substances being set free upon the fermentation, said neutralisation being brought about by means of suitable alkaline mineral salts, such as for instance carbonates.

(2) In the process under claim ONE the use of basic nitrogenous salts.

(3) The use of the process under claim ONE also in the case of such processes of fermentation, where it is not intended to obtain yeast.

DEFENDANT'S EXHIBIT D-6.

Copy of German Patent No. 300,662 of November 12, 1919.

(Translation Only Printed.)

TRANSLATION.

German Patent No. 300,662

Verein der Spiritus Fabrikanten in Deutschland

Issued November 12, 1919—Filed March 17, 1915

"Process for the Production of Yeast Without or With Only Small Production of Alcohol"

In the production of yeast according to the wort aïration process by use of the usual operation considerable quantities of alcohol are obtained the amount of which depending on the amount of sugar which is at the disposal of the yeast. It has been tried to work out suitable processes for the conditions whereby an increase of the yield in yeast from the applied raw materials at the expense of the production of alcohol appears more profitable. It has been suggested, for example, to dilute the worts to such a degree that the alcohol produced from the sugar can again be assimilated as nutrient by the yeast. The same purpose should be obtained by the patent 10,135, Class 6, in such a manner that the nutrients necessary for the yeast (sugar, peptons) are added in portions or continuously according to need and so slowly that the yeast utilizes the sugar not for the formation of alcohol, but only

for the nutrition. Also in this case a very small concentration of sugar, or, in other words, a very far extended dilution is necessary; by the increase of the operation costs connected with this high dilution, however, in both processes the advantage of the high yield of yeast is again partly offset. –

In the process described in the following, forming the matter of the invention, the purpose is obtained of obtaining high yields in yeast without or only with a small production of alcohol by application of worts of normal condition and concentration in such a way that the wort is continuously added to the yeast in such small quantities that it does not form any alcohol in excess, but that it is able to again assimilate it while still forming.

The process is performed, for example, in the following manner:

A wort is produced in the manner as usual in the pressed yeast industry (for example of 12 per cent Balling), a part of the wort is strongly diluted (for example to 1 per cent Balling), yeast is added and then the airation is started in a known manner. The remainder of the concentrated wort is now continuously added in such amounts that not only the alcohol formed from the sugar which was present at the beginning, but also the alcohol which is formed from the sugar which is added together with the wort can at once be assimilated by the yeast. If a total airation of 10 hours is provided the addition is made within 7 to 8 hours with continuous airation, and the airation is continued for 2 to 3 hours without the addition of wort. The quantity of the thin wort which is at the disposal of the yeast at the beginning is, for example, provided in such an amount that the totally applied wort would show an average value of 3 to 5 per cent Balling.

In contradistinction to the process of producing yeast from worts of very small concentration, the

aforementioned process shows the advantage that the yeast is continuously provided with new nutrient by the continuous addition of concentrated wort and that, because of the continuous consumption of the nutrients by the yeast, at the same time, always only such a concentration of the wort is present whereby the nutrients and the continuously formed conversion materials are best and quickest absorbed by the yeast, that means, by the peculiar distribution of the wort the advantages of the great dilution upon normal wort concentration (calculated for the total quantity) are obtained.

In the process according to the patent 10,135 in fact also the periodical or continuous addition of sugar or peptons to the liquid containing the yeast is provided, by utilization of the high dilution which is of advantage for the growth of the yeast at the expense of the formation of alcohol. However, aside from the fact that this process requires a continuous observation of the conditions of the nutrients in the liquid, since sugar and peptons are separately added and only according to demand, the operation with normally concentrated liquids has not been provided; the simple administration of the aforementioned process is therefore missing which does not differ in any way from the normal process of the production of airation yeast in the production of the wort and in the expenditure of work.

The preparation of the first undiluted setting wort with yeast and the addition of the remaining wort which is more and more diluted by the "Nachschwänzen" has been known in the production of pressed yeast. By this operation, which follows the opposite principle, however, such large quantities of alcohol are produced from the very beginning that an assimilation of the alcohol which is worth mentioning cannot arise anymore.

CLAIM:

Process for the production of yeast without or with only a small production of alcohol, characterized in that the cultivation of yeast is started in strongly diluted wort of a known composition and that to the diluted wort a wort of higher concentration is continuously slowly added.

DEFENDANT'S EXHIBIT D-7-1.

Synopses of File Wrapper Contents of United States Patent No. 1,449,103 to Friedrich Hayduck, Dated March 20, 1923.

(Physical Exhibit.)

DEFENDANT'S EXHIBIT D-7-2.

Synopses of File Wrapper Contents of United States Patent No. 1,449,105 to Friedrich Hayduck, Dated March 20, 1923.

(Physical Exhibit.)

DEFENDANT'S EXHIBIT D-7-3.

Synopses of File Wrapper Contents of United States Patent No. 1,449,106 to Friedrich Hayduck, Dated March 20, 1923.

(Physical Exhibit.)

DEFENDANT'S EXHIBIT D-7-4.

Synopses of File Wrapper Contents of United States Patent No. 1,449,109 to Friedrich Hayduck, Dated March 20, 1923.

(Physical Exhibit.)

DEFENDANT'S EXHIBIT D-8-1.

Patented Mar. 20, 1923. 1,449,127

UNITED STATES PATENT OFFICE.

MARTIN NILSSON AND NORMAN S. HARRISON, OF PEEKSKILL, NEW YORK, ASSIGNORS TO THE FLEISCHMANN COMPANY, OF NEW YORK, N. Y., A CORPORATION OF OHIO.

PROCESS FOR PRODUCING YEAST.

No Drawing. Application filed January 7, 1919. Serial No. 270,068.

To all whom it may concern:

Be it known that we, MARTIN NILSSON, a citizen of the United States, and NORMAN S. HARRISON, a subject of Great Britain, residing, respectively, in Peekskill, county of Westchester, and State of New York, have invented new and useful Improvements in Processes for Producing Yeast, of which the following is a full, clear, and exact specification.

Our invention relates to processes for the production of yeast and refers particularly to a process for the production of bakers' yeast from saccharíferous materials, such as molasses.

The object of our invention is the provision of a process for producing yeast with relatively high yields by growing it from seed yeast in a saccharíferous nutrient solution treated with yeast nutrient compounds, such solution being adapted for use in the art of yeast manufacture instead of cereal worts.

The usual procedure heretofore practiced in the production of yeast involved mashing malted cereals with water at certain temperatures in order to obtain a maximum amount of carbohydrates, proteins and mineral matters in solution. The resulting mash or wort was then filtered and the liquid stocked with yeast, after which it was aerated to promote the growth of yeast. The yeast thus produced was separated from the liquid by some suitable means. The process involving this procedure, however, is a comparatively expensive one as it requires a considerable amount of coal, labor and time incident to its proper practice, while the delicacy of the various operations requires careful and expert attention.

The process of our invention avoids these difficulties and presents a simplified procedure whereby a superior grade of yeast can be produced more economically, with greater certainty of results, and with increased yields.

In the practice of the process of this invention, yeast is propagated in a saccharíferous nutrient solution to which has been added compounds containing phosphorus, and nitrogen in yeast assimilable form, the propagation of yeast being allowed to proceed to the fullest desired extent, dependent upon the quality of yeast to be produced.

The compounds containing phosphorus intended to be included in the practice of this invention are those of an inorganic character adapted to yield at least a portion of their phosphorus content during the period of yeast propagation, as, for example, phosphorous acid; phosphoric acid; phosphate of ammonium; phosphates of alkali metals, such as sodium phosphate, or potassium phosphate; and phosphates of alkali earths, as calcium acid-phosphate, or magnesium acid-phosphate.

The compounds containing nitrogen are intended to include those which are adapted to supply inorganic nitrogen in yeast assimilable form; those preferably employed are ammoniacal compounds, as, for example, aqua-ammonia (ammonium hydroxide) and salts of ammonium.

Certain alkaline compounds, in this process, however, are the equivalent of aqua-ammonia when used with the addition of a suitable nitrogenous compound, as, for example, sodium hydrate, to which has been added ammonium phosphate. In the use of ammonium salts it is preferable always to add an alkali, as, for example, a carbonate or a hydrate, in order that free hydroxyl ions may be supplied while neutralizing the deleterious acidity developed during the period of yeast propagation. The second mentioned class of compounds would therefore include alkaline compounds when used in this manner.

The saccharíferous material employed in making the nutrient solution is of a character adapted to be subject to fermentation under the conditions of yeast propagation; among those which it is contemplated using are beet molasses and cane molasses.

Particularly valuable results are obtained by the use of beet molasses with the addition of a phosphate, such as ammonium phosphate, and ammonia; the yeast which is produced has great strength and improved lasting qualities, the cost of propagation being much less than that incident to the production of yeast by the processes heretofore generally employed.

Some yeasts, as is well known, are of such character that when exposed in masses for a short time tend to break down; when this is extreme they tend to pass into a slimy condition. By our process, however, a product is obtained which does not become slimy or deteriorate with age, and has therefore

what has been above referred to as lasting qualities, such qualities insuring that a mass of yeast will sustain its strong condition for a long period, the fermenting power also being retained.

In practicing the process of our invention, consideration of the results obtained appears to indicate that molasses is rich in practically all the materials necessary for yeast nourishment during the period of yeast propagation, with the exception of materials from which yeast assimilable nitrogen and phosphorus may be obtained while yeast is being propagated. Such constituents are of the greatest economical and commercial value in the manufacture of yeast since they allow the production of increased yields of a superior quality of yeast at a cost considerably below that grown from cereal worts. The saccharriferous material here employed is, however, deficient in these constituents, which deficiency is compensated for by the added compounds.

We have found that while yeast may be produced from cane molasses by our process, it is preferable to use beet molasses, as the yeast obtained from it is stronger, whiter, purer, more lasting, more easily manufactured and even less expensive that that produced from cane molasses.

By the process of this invention yeast can be propagated in a nutrient solution containing molasses, whether it be acid or alkaline, during the period of yeast growth, but we prefer that the condition of the nutrient solution be that of approximate neutrality since this condition appears to be conducive to maximum yeast production, though it is known that yeast multiplication is carried on most thoroughly when the nutrient solution is slightly acid. The acidity of the solution, however, if allowed to rise, will soon reach a point where the multiplication of yeast cells is deleteriously effected. On the other hand, if the solution is too strongly akaline there will be a cessation of yeast development, though not necessarily a cessation of the production of alcohol, it being known that the production of alcohol is dependent upon the presence of zymase and is therefore not directly commensurate with yeast multiplication.

The acidity which develops while yeast is multiplying is additive to the slight initial acidity preferably maintained for economic yeast culture, such acidity being produced by the partial assimilation of the salts in solution during the process of yeast propagation, which results in releasing acid radicals or components, as, for example, the partial assimilation of ammonium phosphate by yeast results in releasing the radical of phosphoric acid. It is for the purpose of rebinding the acid radicals thus released so as to restore the original conditions approximating neutrality as far as possible that compounds adapted to supply hydroxyl ions are added to the nutrient solution.

The following is an example of the process of our invention:—

560 pounds of practically neutral beet-molasses are diluted with 950 gallons of water and a solution of 14 pounds of phosphate of ammonium is gradually added. 11.2 pounds of yeast are then introduced. Air is blown into the mixture until the production of yeast is ended, the mixture being maintained in a practically neutral condition by the addition of ammonia from time to time to accomplish this result. The mixture is maintained at a temperature of 25°–30° C. during the operation which usually occupies about 24 hours. The yeast is then separated from the remainder of the mixture in any suitable manner. The process produces from 50%–60% of an extremely strong yeast which is not nearly as subject to deterioration as is the yeast produced in the ordinary way.

While the example given employs only one phosphorus-containing compound, a mixture of several phosphorus containing compounds may be used as a mixture of phosphate of ammonium and acid-phosphate of calcium.

We do not limit ourselves to the particular compounds, quantities, temperatures, duration of reaction or mode of procedure described, all of which may be varied without going beyond the scope of our invention as described and claimed.

Having described our invention, what we claim as new and desire to secure by Letters Patent is:

1. A process of producing yeast, which comprises preparing a yeast nutrient solution, propagating yeast therein with aeration, said yeast nutrient solution containing only molasses and inorganic compounds containing phosphorus and nitrogen, which are adapted to yield at least a portion of their content to the yeast and to set free components which tend increasingly to acidify the solution during propagation, and during the period of propagation neutralizing the excess of such acidity.

2. A process of producing yeast, which comprises preparing a yeast nutrient solution, propagating yeast therein with aeration, said yeast nutrient solution containing only molassses and phosphate of ammonium adapted to be at least partially assimilated by the yeast and to set free components which tend increasingly to acidify the solution during propagation, and during the period of propagation neutralizing the excess of such acidity.

3. A process of producing yeast, which

comprises preparing a yeast nutrient solution, propagating yeast therein with aeration, said yeast nutrient solution containing essentially molasses and inorganic compounds containing phosphorus and adapted to yield at least a portion of their content to the yeast and to set free components which tend increasingly to acidify the solution during propagation, and introducing into the solution during the period of propagation a compound, containing inorganic nitrogen in yeast assimilable form and having an alkaline reaction, in quantities adapted to neutralize the excess of such acidity.

4. A process of producing yeast, which comprises preparing a yeast nutrient solution, propagating yeast therein with aeration, said nutrient solution containing essentially molasses and phosphate of ammonium adapted to be at least partially assimilated by the yeast and to set free components which tend increasingly to acidify the nutrient solution during propagation, and introducing into the solution during the period of propagation a compound, containing inorganic nitrogen in yeast assimilable form and having an alkaline reaction, in quantities adapted to neutralize the excess of such acidity.

5. A process of producing yeast which comprises preparing a yeast nutrient solution, propagating yeast therein with aeration, said yeast nutrient solution containing essentially beet molasses and inorganic compounds containing phosphorus and adapted to yield at least a portion of their content to the yeast and to release components which tend increasingly to acidify the nutrient solution during propagation, and introducing into the nutrient solution an ammoniacal compound, adapted to supply inorganic nitrogen in yeast assimilable form and having an alkaline reaction, in amounts sufficient to neutralize the excess of such acidity.

6. A process of producing yeast which comprises preparing a yeast nutrient solution, propagating yeast therein with aeration, said yeast nutrient solution containing essentially beet molasses and phosphate of ammonium adapted to yield at least a portion of its content to the yeast and to release components which tend increasingly to acidify the nutrient solution during propagation, and introducing into the nutrient solution during the period of propagation aqua-ammonia in amounts sufficient to neutralize the excess of such acidity.

7. In the art of yeast manufacture, the process which comprises preparing in a fermentor a yeast nutrient solution containing essentially beet molasses and inorganic compounds adapted to supply phosphorus and nitrogen and to release components which tend increasingly to acidify the solution during propagation, seeding the solution with yeast, effecting propagation with aeration while maintaining the temperature between the limits of 25° to 30° C., and during the period of propagation neutralizing the excess of such acidity by the addition of an ammoniacal compound having an alkaline reaction.

8. In the art of manufacturing bakers' yeast, the process which comprises preparing a yeast nutrient solution in a fermentor with beet molasses and phosphate of ammonium adapted to supply at least a portion of its content and to release components which tend increasingly to acidify the solution during propagation, introducing seed yeast, effecting propagation with aeration at a temperature between the limits of 25° to 30° C., introducing into the fermentor aqua-ammonia in such quantities as to neutralize substantially the excess of said acidity, and thereafter separating the yeast.

9. A process of producing yeast, which comprises preparing a yeast nutrient solution, propagating yeast therein with aeration, said yeast nutrient solution containing only molasses and an inorganic compound containing phosphorus and nitrogen, which is adapted to yield at least a portion of its content to the yeast and to set free components which tend increasingly to acidify the solution during propagation, and during the period of propagation neutralizing the excess of such acidity.

10. A process of producing yeast, which comprises preparing a yeast nutrient solution, propagating yeast therein with aeration, said yeast nutrient solution containing essentially molasses and an inorganic compound containing phosphorus and adapted to yield at least a portion of its content to the yeast and to set free components which tend increasingly to acidify the solution during propagation, and introducing into the solution during the period of propagation a compound, containing inorganic nitrogen in yeast assimilable form and having an alkaline reaction, in quantities adapted to neutralize the excess of such acidity.

11. A process of producing yeast which comprises preparing a yeast nutrient solution, propagating yeast therein with aeration, said yeast nutrient solution containing essentially beet molasses and an inorganic compound containing phosphorus and adapted to yield at least a portion of its content to the yeast and to release components which tend increasingly to acidify the nutrient solution during propagation and introducing into the nutrient solution an ammoniacal compound, adapted to supply inorganic nitrogen in yeast assimilable form and having an alkaline reaction, in amounts sufficient to neutralize the excess of such acidity.

12. In the art of manufacturing bakers' yeast a step which comprises preparing a yeast nutrient solution, adapted for large commercial yields of yeast, consisting essentially of water, molasses and an inorganic compound containing phosphorus and nitrogen in yeast assimilable form, in amounts sufficient to supply all essential yeast nutrients during the propagating period.

13. In the art of manufacturing bakers' yeast the step which comprises preparing a yeast nutrient solution, adapted for large commercial yields of yeast, consisting of essentially water, molasses and an admixture of yeast nourishing inorganic compounds containing prosphorus and nitrogen in yeast assimilable form, in amounts sufficient to supply all essential yeast nutrients during the propagating period.

14. In the art of manufacturing bakers' yeast the step which comprises preparing a yeast nutrient solution, adapted for large commercial yields of yeast, consisting of essentially water, beet molasses and ammonium phosphate, in amounts sufficient to supply all esential yeast nutrients during the propagating period.

Signed at New York city, in the county of New York and State of New York, this 3rd day of January, 1919.

MARTIN NILSSON.
NORMAN S. HARRISON.

DEFENDANT'S EXHIBIT D-8-2.

Patented Mar. 20, 1923. 1,449,134

UNITED STATES PATENT OFFICE.

ALFRED WOHL, OF DANZIG-LANGFUHR, FREE CITY OF DANZIG, ASSIGNOR TO THE FLEISCHMANN COMPANY, OF NEW YORK, N. Y., A CORPORATION OF OHIO.

METHOD OF TREATING MOLASSES.

No Drawing. Application filed August 31, 1921. Serial No. 497,323.

To all whom it may concern:

Be it known that I, ALFRED WOHL, residing at Danzig-Langfuhr, Free City of Danzig, have invented certain new and useful Improvements in Methods of Treating Molasses (for which applications for patent were filed as follows: France, filed on June 11th, 1920; Denmark, filed on June 11th, 1920; United States of America, filed on June 23d, 1920; Czechoslovakia, filed on June 25th, 1920; Canada, filed on July 10th, 1920; Spain, filed on July 14th, 1920; Rumania, filed August 6th, 1920; Germany, filed April 6th, 1920), of which the following is a specification.

This invention relates to a method of treating molasses with a view to its later utilization in the fermentation industries and more particularly relates to a method of clarifying molasses while in a fairly concentrated state and by heating only to a relatively low temperature, with a view to the propagation of yeast of good keeping qualities, color and baking strength, therein.

An object of this invention is to accomplish such clarification and sterilization of molasses by an efficient method which is commercially practicable and easily carried out with a minimum of time, labor and cost.

Another object is to obviate the long, expensive, and tedious boiling commonly practiced.

Other objects will be in part obvious and in part pointed out hereinafter.

The invention accordingly consists in the several steps and the relation and order of one or more of such steps with respect to each of the others thereof, which will be exemplified in the hereinafter disclosed method and the scope of the application of which will be indicated in the claims that follow.

The methods heretofore employed for producing compressed yeast from molasses are based upon the assumption previously existing that a boiling of an acidulated solution of molasses is necessary, both for the purpose of inverting the sugar and separating the constituents difficult of solution, and for sterilizing the molasses. Although the first effect, that of inverting the sugar, is accomplished by boiling acidulated molasses, it is known that sugars are inverted by certain enzymes in yeast. However, for the purpose of sterilization, boiling has been considered to be essential.

I have found that compressed yeast of good keeping qualities and answering all requirements of a good bakers' yeast can be produced from molasses and ammonium salts without further organic nitrogenous foods, without lactic acid fermentation or the addition of lactic acid, and also without boiling, if the molasses, in a moderately concentrated solution, for instance, 30 to 40° Brix, is mixed with large quantities of strong mineral acid and allowed to stand at ordinary temperature or at a gentle heat until sterilization is accomplished. This should be done before any further dilution in order not to use unnecessarily large quantities of acid, which would be required to bring the degree of acidity up to the desired amount, and which would later necessitate the neutralization of a large portion of the acid.

As an example, the quantity of mineral acid necessary, is that which will be sufficient to acidify the subsequent solution of molasses up to the strength needed for producing yeast of good quality without souring by the addition or formation of lactic acid. For instance, I find that to every 100 parts by weight of molasses, about 2 parts by volume of concentrated sulphuric acid, which has been preferably previously diluted with a little water, should be used. The difference as compared with the method of acidulation heretofore practiced lies in the fact that these quantities of acid are, in the form of a strong mineral acid, e. g. sulphuric acid or hydrochloric acid, added to the molasses in a relatively high concentration and after they have exercised their sterilizing effect a suitable dilution is effected.

My method of causing the quantities of acid which will afterwards be required for acidifying the highly diluted fermentative or yeast wort, to act prior to the final dilution of the molasses also allows a further step which improves the hitherto exceedingly inefficient utilization of the mineral phosphoric acid and phosphates as yeast foods.

If phosphoric acid is added, as is usually the case, as super-phosphate, a considerable proportion of the phosphoric acid always

remains unused. I overcome this inefficiency by first strongly heating together the super-phosphate and the mineral acid intended for the acidulation of molasses, then adding the mixture to the molasses and heating in the manner hereinbefore described, whereby the molasses is not only sterilized but phosphoric acid is obtained in a form which is readily assimilable by yeast.

When super-phosphate and sulphuric acid are treated as above described, gypsum (calcium sulphate) is formed by their reaction and this is later disseminated throughout the acidified solution of molasses. Since calcium sulphate readily combines with sulphate of potassium to form calcium-potassium sulphate, the calcium sulphate combines with the potassium salts present in the molasses to form insoluble calcium-potassium sulphate which accordingly separates from the molasses and accomplishes in a simple and efficient manner, the reduction of the amount of potassium salts in the molasses. This decrease or reduction in the amount of potassium salts present in the molasses may be increased by the addition of more gypsum to the concentrated and acidulated molasses.

Moreover when super-phosphate is treated with sulphuric acid as above described, some calcium sulphate is precipitated out, and this precipitate may be separated from the liquid if desired, but preferably the liquid and precipitate are both added to the molasses whereby the clarifying effect of the precipitate is utilized. In the former case, special measures are necessary for the practical clarification of the molasses solution. This may be done by allowing the bulk of the deposit to settle and filtering the pre-clarified solution through a flat filter or a sand filter. From the constituents rich in slime, there are then obtained, by means of a centrifugal separator, further quantities of liquid, poor in slime and hence easily filtered, which liquid is then clarified in the above manner. The mud residue can be drawn off with acidulated water and centrifuged or can be used for the manufacture of spirit without the production of yeast.

In order to test the sufficiency the above method, separate portions of molasses were inoculated with traces of two mildew fungi (Mucor and Aspergillus) and two bacteria (*Bacterium lactis* and *butyricum*), and allowed to stand for 8 days at 25° C. From these portions a solution of 30° Brix was made at ordinary temperature, with the addition of acid as indicated; a second solution without an addition of acid was also taken. These solutions were tested after 24 hours, as to the number of micro-organisms present therein, by inoculation upon nutrient gelatine. The first solution was found to be entirely sterile whereas the second solution contained on the average of 1800 organisms per cubic centimeter.

As many changes could be made in carrying out the above method without departing from the scope of the invention, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

It is also to be understood that the following claims are intended to cover all of the generic and specific features of the invention herein described and all statements of the scope of the invention, which, as a matter of language, may be said to fall therebetween.

This application is a continuation of certain subject-matter originally disclosed in my co-pending application, Serial No 391,156, filed June 23, 1920.

Having described my invention, what I claim as new and desire to secure by Letters Patent is:

1. The method of treating molasses which comprises adding thereto a mineral acid and a phosphate.

2. The method of treating molasses which comprises, adding thereto a mineral acid and a phosphate, and heating the mixture moderately.

3. The method treating molasses which comprises, adding thereto a mixture of a mineral acid and a phosphate, heating the final mixture until a precipitate is formed, and separating the precipitate from the liquid.

4. The method of preliminary treatment of molasses for use in the fermentation industries which comprises, treating a phosphate with an excess of mineral acid, heating the same, adding the resultant mixture to the molasses, and heating the final mixture until a precipitate is formed.

5. The method of preliminary treatment of molasses for use in the fermentation industries which comprises, treating a phosphate with an excess of mineral acid, heating the same, adding the resultant mixture to the molasses, heating the final mixture until a precipitate is formed, and separating the precipitate from the liquid.

6. The method of preliminary treatment of molasses for use in the fermentation industries which comprises, treating a phosphate with an excess of a mineral acid, and adding the resultant mixture to the molasses.

7. The method of preliminary treatment of molasses for use in the fermentation industries which comprises, treating a phosphate with an excess of a mineral acid, heating the same, and adding the resultant mixture to the molasses.

8. The method of preliminary treatment

of molasses for use in the fermentation industries which comprises, heating an excess of mineral acid with a phosphate until a precipitate is formed, adding the mixture to the molasses, and heating moderately.

9. The method of preliminary treatment of molasses for use in the fermentation industries which comprises, heating an excess of mineral acid with a phosphate until a precipitate is formed, separating the precipitate from the liquid, adding the liquid to the molasses, and heating the final mixture moderately.

10. The method of preliminary treatment of molasses for use in the fermentation industries which comprises, heating an excess of mineral acid with a super-phosphate until a precipitate is formed, and adding the mixture to the molasses.

11. The method or preliminary treatment of molasses for use in the fermentation industries which comprises, heating an excess of mineral acid with a super-phosphate until a precipitate is formed, adding the mixture to the molasses, and heating moderately.

12. The method of preliminary treatment of molasses for use in the fermentation industries which comprises, heating an excess of mineral acid with a super-phosphate until a precipitate is formed, separating the precipitate from the liquid, and adding the liquid to the molasses.

13. The method of preliminary treatment of molasses for use in the fermentation industries which comprises, heating an excess of mineral acid with a super-phosphate until a precipitate is formed, separating the precipitate from the liquid, adding the liquid to the molasses, and heating the final mixture moderately.

14. In the art of yeast manufacture, the steps in the preparation of a yeast nutrient solution which comprise, preparing a moderately concentrated solution of molasses, treating a super-phosphate with a mineral acid, heating the latter mixture until a precipitate is formed, adding the resultant mixture to the molasses solution, moderately heating the final mixture until a precipitate is formed, and separating the precipitate from the liquid.

15. In the art of yeast manufacture, the steps in the preparation of a yeast nutrient solution which comprise, preparing a moderately concentrated solution of molasses, adding thereto a mixture of a mineral acid and a phosphate, and treating the resultant mixture in such a manner that a portion of the mineral salts, originally present in the molasses, combine with the materials added to the molasses to form an insoluble salt which may be readily separated from the molasses.

16. In the art of yeast manufacture, the steps in the preparation of a yeast nutrient solution which comprise, preparing a moderately concentrated solution of molasses, adding thereto a mixture of a mineral acid and a phosphate, heating the resultant mixture, whereby a portion of the mineral salts originally present in the molasses, combine with the mixture added to the molasses to form an insoluble salt, and to set phosphoric acid free in a form which is readily assimilable by yeast, and separating the precipitate from liquid.

17. In the art of yeast manufacture, the steps in the preparation of a yeast nutrient solution which comprise, preparing a moderately concentrated solution of molasses and adding thereto such an excess of mineral acid, as will upon final dilution, produce in the resultant yeast nutrient solution an acidity which approximates the optimum for the yeast propagation.

18. In the art of yeast manufacture, the steps in the preparation of a yeast nutrient solution which comprise, preparing a moderately concentrated solution of molasses and adding thereto a phosphate and such an excess of mineral acid, as will upon final dilution produce in the resultant yeast nutrient solution an acidity which approximates the optimum for the yeast propagation.

19. The method of preliminary treatment of molasses for use in the fermentation industries which comprises, acidifying the molasses, adding gypsum thereto, and after a precipitate is formed, separating the precipitate from the liquid.

20. The method of preliminary treatment of molasses for use in the fermentation industries which comprises, acidifying the molasses, adding gypsum thereto, and moderately heating the resultant mixture until a precipitate is formed.

In testimony whereof I affix my signature in presence of two witnesses.

ALFRED WOHL.

Witnesses:
HOWARD A. BOURMAN,
WILLIAM M. ROBERTSON.

DEFENDANT'S EXHIBIT D-8-3.

Patented Nov. 27, 1923. 1,475,215

UNITED STATES PATENT OFFICE.

ALFRED WOHL, OF DANZIG-LANGFUHR, FREE CITY OF DANZIG, ASSIGNOR TO THE FLEISCHMANN COMPANY, OF NEW YORK, N. Y., A CORPORATION OF OHIO.

PROCESS FOR THE MANUFACTURE OF YEAST.

Continuation of applications filed June 23, 1920, Serial No. 391,156, and August 31, 1921, Serial No. 497,223. This application filed August 31, 1921. Serial No. 497,221.

(GRANTED UNDER THE PROVISIONS OF THE ACT OF MARCH 3, 1921, 41 STAT. L., 1313.)

To all whom it may concern:

Be it known that I, ALFRED WOHL, residing at Danzig-Langfuhr, Free City of Danzig, have invented certain new and useful Improvements in the Process for the Manufacture of Yeast (for which application for patent were filed as follows: France, filed June 11, 1920; Denmark, filed June 11, 1920; United States of America, filed June 23, 1920; Czechoslovakia, filed June 25, 1920; Canada, filed June 10, 1920; Spain, filed July 14, 1920; Rumania, filed August 6, 1920; Germany, filed January 15, 1915, Patent 310580; Sweden, filed December 29, 1919; Poland, filed June 7, 1920), of which the following is a specification.

This invention relates to the production of yeast and more particularly to the production of yeast in a nutrient wort which is made from cereal material in the usual proportions except that the amount of organic nitrogen usually used is materially reduced, and the missing yeast-assimilable nitrogen is replaced by an inorganic compound containing yeast-assimilable nitrogen.

An object of this invention is the production of compressed yeast by a process which is adapted to be easily carried out, producing a large yield of yeast and materially lowering the cost of production of the yeast as compared with that produced in the usual cereal wort process.

Another object of the invention is the production of yeast from a nutrient solution containing essentially sacchariferous material and other yeast nutrients including cereal material and inorganic nitrogen containing compounds adapted to be at least partially assimilated by the yeast and during such assimilation to set free components which tend increasingly to acidify the nutrient solution, the yeast-assimilable organic nitrogen contained in the cereal material being present to an appreciably lesser degree than in a usual cereal wort, and the missing yeast assimilable nitrogen being added in the form of inorganic compounds. The propagation is carried on with aeration, and at least a substantial portion of the released acidity is neutralized at the election of the operator, the yeast obtained from this wort, either with or without neutralization, being capable of yielding sturdy successive generations indefinitely in continuous operation, that is, without revivification.

Other objects will be in part obvious and in part pointed out hereinafter.

The invention accordingly consists in the several steps and the relation and order of one or more such steps with relation to each of the others thereof, which will be exemplified in the hereinafter disclosed process and the scope of the application of which will be indicated in the claims that follow.

The nitrogenous substances which are used for nutrition in the growth of yeast can emanate from very different sources. Generally, amino nitrogen is considered the most favorable for fermentation and for the increase of the yeast. In the practical manufacture of aerated yeast, malt sprouts are generally used in addition to malt as a source of nitrogenous food. The indefinitely continued commercial production of yeast of fully satisfactory quality in successive generations without revivifications, with inorganic salts of ammonia as the sole nitrogen source is not possible. Experiments show that the yield and fermentative power fall with exceeding rapidity to values which are of no use in practice as soon as the excess of organically bound nitrogen contained in the seed yeast is expended.

The process hereinafter described is based on the new and unexpected observation that a partial substitution of yeast-assimilable organic nitrogen by the cheaper inorganic ammonium salts, up to the extent indeed of nearly one-half, has practically no influence on the yield of yeast and the fermentative power of the yeast produced. Beyond this limit a noticeable reduction, which grows larger in proportion as the organic nitrogen decreases, takes place in the yield of yeast, as is shown by the accompanying curve representing the results of experiments, this being especially true unless at least a substantial portion of the acidity, which results from a partial assimilation of the inorganic ammonium salts and the consequent release of components from such

salts which tend to produce an increasing acidity, be neutralized.

We are here dealing with an effect in which the organic nitrogen compounds and the ammonium salts mutually contribute to the nutrition of the yeast. For example, if in an experiment the ammonium salts are omitted, then under otherwise similar conditions instead of 4.6, only 2.99 grams of dry substance will be obtained, in the shape of newly formed yeast, per 1 litre of 2½% sugar solution.

In the experiments shown in the curve a total concentration of nitrogen was used sufficient for a maximum yield by weight, but not sufficient to keep the newly produced yeast up to the nitrogen content of the seed yeast, for in that case the total quantity would as in actual practice, have to be increased about 2½ to 3 times. Then again it is found however that a mixture in the proportion of 40 to 50% of the total yeast-assimilable nitrogen in the form of ammonium salts is entirely sufficient to allow of breeding six or more generations of yeast in succession without the yield or proportion of nitrogen falling off more rapidly than would be the case in actual practice where the same content of nitrogen is offered by malt sprouts and malt. Indeed according to the present invention the germinating power is even better preserved than under known working conditions.

With a view to improving the properties of the yeast, it is found useful to supply the ammonium salts in two or more stages. This does not influence the yield, but imparts a better compressibility to the yeast.

From the summary of the literature by Pringsheim published in the Wochenschrift f. Brauerei 1907, page 126, it is known that, qualitatively, yeast can take up nitrogen as a food in the form of ammonia both from solutions containing ammoniacal nitrogen (Bokorny) and from solutions containing ammoniacal nitrogen mixed with asparagin (Petit.) but it is also known that with ammoniacal nitrogen alone, it can not be so fed that it remains practically useable. This is also confirmed by our experiments. The summary of literature also sets forth that ammoniacal nitrogen is taken up by yeast in the same manner as is asparagin nitrogen (Petit.) the former, at the optimum temperature, even to a large extent (Laborde.) but that the yield of yeast in these nutrients drops from peptone to asparagin (Kussorow) and to ammonium sulphate (Bokorny). This latter observation is also confirmed by our experiments and precludes a direct practical utilization of all these data for the feeding of yeast.

French Patent No. 343,133, which relates to improvements in the course of fermentation, not to the production of yeast, is based upon the use of extract of yeast as nitrogenous food. Apart from this it recommends an addition of inorganic mineral salts (SO_3, P_2O_5, CaO, MgO). Among other phosphates such as potassium phosphate there is mentioned phosphate of ammonia. This equivalency to potassium phosphate as well as the indefinite amounts prescribed, prove that the ammonium salt is here only mentioned as a well-known kind of phosphoric acid food. The same applies to British patent specification No. 21,011/1896 and to the publication concerning the same matter in the Wochenschrift f. Brauerei 1898, p. 237, from which it can be seen that as an origin of nitrogen, maltopeptone is used, and in this only 6% of the nitrogen is present as ammoniacal nitrogen.

It has also been shown that ammonia salts increase the nitrogenous contents and the germinating power of yeast. (see Zeitschrift für Spiritus-Industrie 1910, supplement to No. 11, page 3, col. 1, observations 4 and 5,) but the adding of ammonia salts hitherto has been done in comparatively small quantities only, and it was not realized that it would be possible to work with an exceedingly low concentration of organic nitrogenous substances as compared to the concentration of carbohydrates.

The statement in the Jahrbuch des Vereins der Spiritus-Fabrikanten in Deutschland, second year, (1911 p. 23, paragraph 1) that ammonium salts are utilized by yeast in large quantities for building up its body substance, contains nothing about the conditions under which this is possible in order to have the most favorable yield with reference to the total nitrogen used. From a closer description of experiments in this relation, it is found that in the reaction filter the assimilation of nitrogen by yeast from a sugar solution containing ammonium sulphate, drops from roughly 30% to roughly 7% (Jahrbuch des Vereins der Spiritus-Fabrikanten in Deutschland, 1912, page 37, paragraph 2). Further results of these works are also recorded in the same Jahrbuch, 1913, at the bottom of page 33 and the top of page 34 where the following summary of results achieved are summarized as follows:

1. That ammonium salts cannot even remotely be regarded as complete nitrogen food for yeast.

2. That on the contrary a mixture of amino acids such as is obtained, for instance, from autodigestion of yeast, ensures full nutrition and probably a better nutrition than the albumen, albumose, and peptone feeding such as is presented by use of malt liquor (wort.)

Neither the more favorable utilization of the nitrogen, which is obtained in a suitably proportioned mixture of organic nitro-

gen and ammonia nitrogen, the longer inheritance of the good properties of the yeast produced under these conditions, nor yet the good keeping quality and the influence on pressing qualities of yeast which is produced by the gradual addition of ammonium salts, have hitherto been recognized. All these points, however, are of importance in the practical application of the hereinafter described process of the manufacture of yeast, with aeration, capable of yielding sturdy successive generations indefinitely without revivification and are in striking contrast to experiments which have been carried out in the past and the object of which was only to enrich the wash with albuminous substances and in which no consideration was taken of the properties of yeast which are highly desirable in the commercial compressed yeast manufacturing industry.

Moreover it might be presumed that, with the ordinary organic nitrogenous food, if a comparatively small quantity of ammoniacal nitrogen were supplied in addition, the properties of the yeast would not be altered to any appreciable extent and perhaps also that a quantitatively inconsiderable substitution would not have any effect on the yield of yeast. On the other hand the perception of the fact which is of importance from the point of view of the practical invention, that a substitution of organic nitrogen by ammoniacal nitrogen is possible to so large an extent that a practical advantage results without impairment of the properties of the yeast, its fermenting powers, further propagating powers, or its yield, and that such substitution, has a favorable influence of these properties, is a new discovery.

Although in Example I, which appears hereinafter, an example of that what I have termed a "usual cereal wort" is set forth, it is to be understood that, since the various components of different cereal materials vary within quite wide limits, the formula given is merely an example and may be correspondingly varied in the manner well known to yeast manufacturers. It is pointed out, however, that although the green malt contains a certain percentage of yeast-assimilable organic nitrogen, the malt sprouts contain by far the larger part of the yeast-assimilable organic nitrogen and that, therefore, if in any cereal wort containing approximately the usual amount of yeast-assimilable organic nitrogen supplying materials, a considerable portion thereof be omitted and ammonium salts be added to replace the yeast-assimilable organic nitrogen thus omitted, such replacement of yeast-assimilable organic nitrogen by yeast-assimilable inorganic nitrogen would fall within my disclosure provided that the replacement was more than ten per cent, and did not greatly exceed fifty per cent. The remaining examples, II to V inclusive, show typical compositions of various worts in which varying percentages of yeast-assimilable organic nitrogen have been replaced by yeast-assimilable inorganic nitrogen.

Furthermore, although in Example I the use of no neutralizing agent has been suggested, for the reason that I have found it possible to attain desirable results without the use of such neutralizing agent, however, if as is shown in Example II, not only is a material portion of the organic nitrogen contained in the malt sprouts omitted, but also a considerable portion of the cereals containing saccharifiable matter is omitted, at least a part of the sugars so omitted being replaced by the addition of sugar, I have found it desirable to neutralize at least a substantial portion of the acidity formed in the solution as a result of the partial assimilation of the ammonium salts by the yeast, by incorporating with the wort a relatively insoluble innocuous antacid carbonate or a mixture of such carbonates, such as calcium or magnesium carbonates, adapted progressively to neutralize the released acidity. Although no such addition of a neutralizing agent has been shown in Example I, yet, as has been pointed out above, even though it is not necessary, such may be added at the election of the operator and is therefore comprehended within my invention.

The systematic experiments which had led to these discoveries have been based upon the principle of ascertaining whether a practically useful yeast, in a yield which is commercially practicable could be obtained with a mixed food when it is known that such a yeast and such yields could not be obtained by a process wherein the nitrogenous food is supplied only in the form of ammoniacal salts. That this could be done to practical advantage was the unforeseen result applying to the special case of the production of yeast by the aeration process.

As illustrative of the method of carrying out my invention the following examples are given:

I. In a usual mash composed of 40% malt sprouts, 20% green malt and 40% maize comprising a total mash of 100 hundredweights only 10 hundred-weights of malt sprouts are used and the nitrogen which is usually supplied by the other 30% of malt sprouts is supplied in the form of ammoniacal salts, for example, 150 kilograms of ammonium sulphate or a corresponding amount of ammonium phosphate $(NH_4)_2HPO_4$, ammonium nitrate or other ammonium salt or mixture of salts. The cereals are mashed in the usual manner and the resulting wort is seeded with eight per cent of seed yeast and aerated for about 10

hours, the ammonium salts being gradually added during the propagating period. After the propagation is completed, the yeast is separated and packed in suitable manner

II. A mash of 90 kilograms of cane sugar and 45 kilograms of germinated malt is mashed and the resultant wort is seeded with 15 to 20 kilograms of seed yeast, there being gradually added 9 kilograms of ammonium sulphate, 1½ kilograms of superphosphate and 2½ kilograms each of calcium carbonate and magnesium carbonate. The yield after deducting the seed yeast is 65 kilograms yeast with 54% albumen in the dry substance and 30 litres of spirit. This is 48.1% a yeast and 22.2% spirit, totalling 70.3% relatively to the cane sugar; or 34.4% yeast and 18.2% spirit, totalling 57.6% as referred to the grain used.

III. A mash of 90 kilograms of cane sugar, 25 kilograms of germinated malt, 2½ kilograms of ammonium sulphate, 0.2 kilograms of magnesium sulphate and 2½ kilograms of calcium carbonate is treated as above and from the same, after deducting the seed yeast, there is obtained 39 kilograms of yeast and 40 litres of spirit. This makes 33.9% yeast and 34.8% spirit, totalling 68.7% relatively to the cane sugar; or 26.8% yeast and 27.6% spirit, totalling 54.4% referred to the grain used.

IV. A mash of 900 kilograms of green malt, 500 kilograms of germinated malt and 3200 kilograms of cane sugar (combined mash of a weight of 5140 kilograms) is started with 400 kilograms of yeast, with a gradual addition of 60 kilograms of ammonium sulphate and 60 kilograms of calcium carbonate, and are aerated in the usual way. There is obtained net 1740 kilograms of good and well pressable yeast and 1620 litres of spirit. This makes 40.5% of yeast and 37.6% of spirit, totalling 78.1% relatively to cane sugar; 33.8% yeast and 31.5% spirit, totalling 65.3% relatively to the grain used. According to Examples (III) and (IV), one and the same yeast was carried forward for months in a distillery.

V. In a further working experiment according to Example IV, but without the addition of ammonium sulphate and with addition of 400 kilograms of dry spent grain, there was obtained a net yield of 1410 kilograms yeast and 1620 litres of spirit. This makes 32.7% yeast and 37.6% spirit, totalling 70.3% relatively to cane sugar: or 27.4% yeast and 31.5% spirit, totalling 58.9% relatively to the grain used. The spent grain added for filtering has not been taken into account at all in calculating the yield relative to the grain used otherwise the difference would be a little greater still.

Example IV cannot be directly compared with Examples II and III because in the Example IV green malt also is used and this supplies nitrogen in a form which is readily assimilated by yeast.

As certain changes could be made in carrying out the above process without departing from the scope of the invention, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

This case is a continuation of my co-pending application, Serial No. 391,156 filed June 23, 1920, and a continuation of and a consolidation with my co-pending application, Serial No. 497,322 filed August 31, 1921.

Having described my invention, what I claim as new and desire to secure by Letters Patent, is:

1. The process of manufacturing yeast which comprises preparing a cereal wort in which the yeast-assimilable organic nitrogen is replaced to an amount of from 10% to 50% by yeast-assimilable ammoniacal nitrogen, and propagating yeast therein with aeration.

2. The process of manufacturing yeast which comprises preparing a cereal wort including an amount of yeast-assimilable organic nitrogen which is less, by from 10% to 50%, than the amount normally present in a usual cereal wort, supplying the deficiency of yeast-assimilable organic nitrogen by adding inorganic salts containing yeast-assimilable nitrogen, and propagating yeast therein with aeration.

3. The process of manufacturing yeast which comprises preparing a cereal wort including an amount of yeast-assimilable organic nitrogen which is less, by from 10% to 50%, than the amount normally present in a usual cereal wort, propagating yeast therein with aeration, and during the process of propagation supplying the deficiency of yeast-assimilable nitrogen by the fractional addition of an inorganic compound containing yeast-assimilable nitrogen.

4. The process of manufacturing yeast which comprises preparing a yeast nutrient solution, containing essentially sacchariferous material and other yeast nutrients, including cereal material and an inorganic compound containing yeast-assimilable nitrogen substituted for organic nitrogen normally present in a usual cereal wort to an extent whereby the yeast is capable of yielding sturdy successive generations indefinitely in continued operation, and propagating yeast therein with aeration.

5. The process of manufacturing yeast capable of yielding sturdy successive generations indefinitely in continued operation, which comprises preparing a yeast nutrient solution, containing essentially sacchariferous material and other yeast nutrients including cereal material present only in such

amounts as will cause the yeast nutrient solution to contain from 10% to 50% less than the amounts of yeast-assimilable organic nitrogen normally present in a usual cereal wort and including an inorganic compound containing yeast-assimilable nitrogen in amounts sufficient to supply the deficiency of yeast-assimilable nitrogen, and propagating yeast therein with aeration.

6. The process of manufacturing yeast capable of yielding sturdy successive generations indefinitely in continued operation, which comprises preparing a yeast nutrient solution containing essentially saccharifer-ous material and other yeast nutrients, including cereal material present only in such amounts as will cause the yeast nutrient solution to contain from 10% to 50% less than the amounts of yeast-assimilable organic nitrogen normally present in a usual cereal wort, propagating yeast therein with aeration, and during the process of propagation supplying the deficiency of yeast-assimilable nitrogen by the fractional addition of inorganic compounds containing yeast-assimilable nitrogen.

7. The process of manufacturing yeast which comprises preparing a cereal wort in which the yeast-assimilable organic nitrogen is replaced to an amount from 10% to 50% by yeast-assimilable ammoniacal nitrogen containing compounds from which components are liberated which tend increasingly to acidify the wort during propagation, adding to the wort sufficient amounts of a relatively insoluble innocuous antacid carbonate to neutralize at least a substantial portion of such acidity, and propagating yeast therein with aeration.

8. The process of manufacturing yeast which comprises preparing a cereal wort including an amount of yeast-assimilable organic nitrogen which is less by from 10% to 50% than the amount normally present in a usual cereal wort, supplying the deficiency of yeast-assimilable nitrogen by adding inorganic salts containing yeast-assimilable nitrogen and from which components are liberated which tend increasingly to acidify the wort during propagation, adding to the wort sufficient amounts of a relatively insoluble innocuous antacid carbonate to neutralize at least a substantial portion of such acidity, and propagating yeast therein with aeration.

9. The process of manufacturing yeast capable of yielding sturdy successive generations indefinitely in continued operation, which comprises preparing a yeast nutrient solution containing essentially saccharifer-ous material and other yeast nutrients, including cereal material present only in such amounts as will cause the yeast nutrient solution to contain from 10% to 50% less than the amounts of yeast-assimilable organic nitrogen normally present in a usual cereal wort and including an inorganic compound containing yeast-assimilable nitrogen in amounts sufficient to supply the deficiency of yeast-assimilable nitrogen and from which components are liberated which tend increasingly to acidify the wort during propagation, adding to the wort sufficient amounts of a relatively insoluble innocuous antacid carbonate to neutralize at least a substantial portion of such acidity, and propagating yeast therein with aeration.

10. The process of manufacturing yeast capable of yielding sturdy successive generations indefinitely in continued operation which comprises preparing a yeast nutrient solution containing essentially saccharifer-ous material and other yeast nutrients, including cereal material present only in such amounts as will cause the yeast nutrient solution to contain from 10% to 50% less than the amounts of yeast-assimilable organic nitrogen normally present in a usual cereal wort, adding to the wort a relatively insoluble innocuous antacid carbonate, propagating therein yeast with aeration, and during the process of propagation supplying the deficiency of yeast-assimilable nitrogen by the fractional addition of an inorganic compound containing yeast-assimilable nitrogen, said insoluble innocuous antacid carbonate being present in amounts sufficient to neutralize at least a substantial portion of such acidity.

In testimony whereof I affix my signature in presence of two witnesses.

ALFRED WOHL.

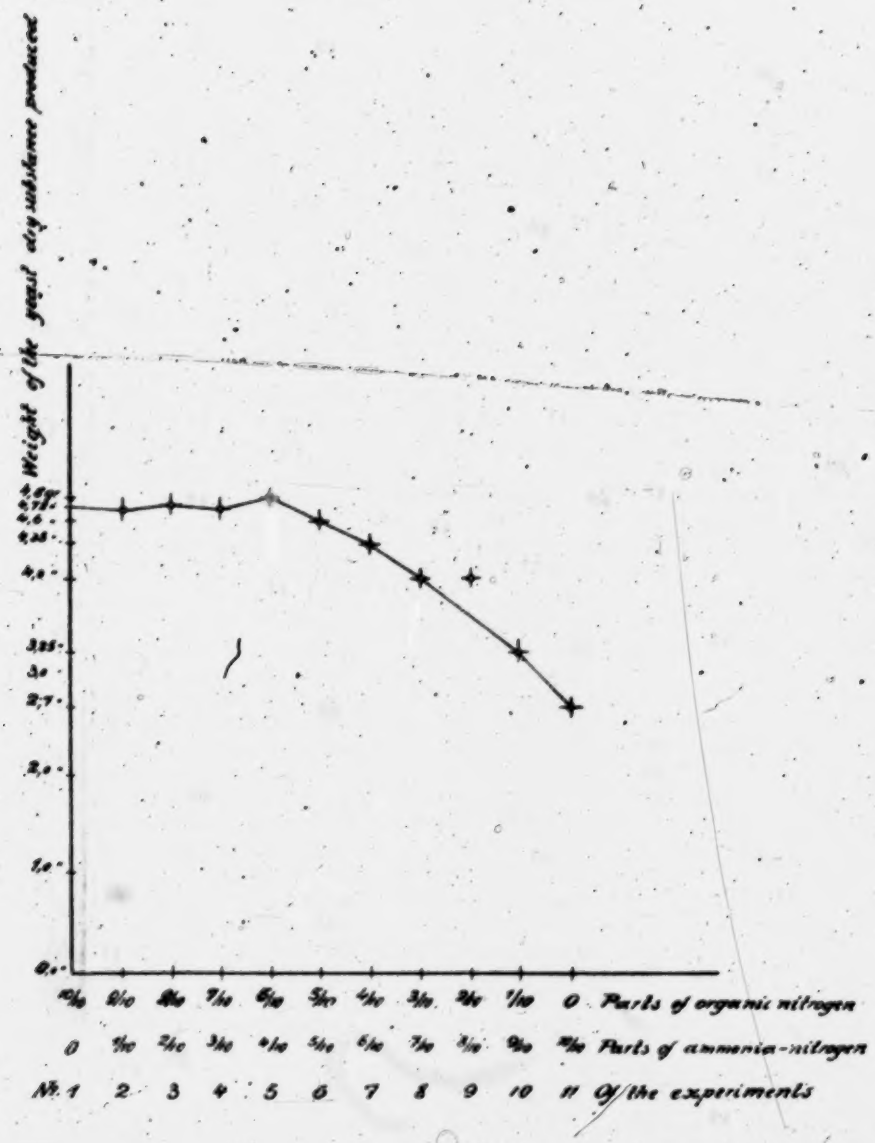
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Nov. 27, 1923. 1,475,215

A. WOHL

PROCESS FOR THE MANUFACTURE OF YEAST

Filed Aug. 31, 1921



INVENTOR

Alfred Wohl

BY E. T. Regan

ATTORNEY

DEFENDANT'S EXHIBIT D-8-4.

Patented Oct. 23, 1928. 1,688,831

UNITED STATES PATENT OFFICE.

FRED RANSCHOFF, OF NEW YORK, N. Y., ASSIGNOR TO THE FLEISCHMANN COMPANY, OF NEW YORK, N. Y., A CORPORATION OF OHIO.

METHOD OF TREATING MOLASSES.

No Drawing. Application filed May 11, 1925. Serial No. 29,623.

This invention relates to the treatment of impure sacchariferous solutions for use in the fermentation industries and more particularly to the clarification of molasses for use in the manufacture of yeast and has as its general object the carrying out of such treatment in a more efficient and economical manner than heretofore.

A more particular object of the present invention is the treatment of cane molasses as the latter is usually commercially obtained in bulk, or a mixture thereof with other sacchariferous substances for the purpose of removing from the molasses certain substances ordinarily present therein which tend seriously to interfere with its use in processes of manufacturing bakers' yeast and to cause the yeast produced therefrom to be of inferior quality.

A further object of the invention is so to carry out the above treatment or clarification that the sacchariferous material is simultaneously enriched with yeast-assimilable substances of such nature and in such quantities as materially to augment its value in yeast production.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

The invention accordingly comprises the several steps and the relation and order of one or more of such steps with respect to each of the others thereof, which will be exemplified in the method hereinafter disclosed, and the scope of the application of which will be indicated in the claims.

Although sacchariferous yeast-nutrient solutions have in recent years been employed successfully in commercial production of bakers' yeast, utilizing both beet and cane molasses as the principal or exclusive source of sugars, available for the yeast growth, more or less difficulty has been experienced in obtaining uniform results as to quality and yield of yeast, especially when cane molasses is used as a source of sugar material.

Cane molasses as it is ordinarily obtained on the market is unsuitable for fully satisfactory use in most present day processes of yeast production for the reason that it usually contains comparatively large amounts of the heavier metals such as iron, aluminum, magnesium and calcium, and is also contaminated with considerable quantities of dirt, sand and various colloidal and pectine substances of unknown composition. Some of these substances are detrimental from the standpoint of the yeast manufacturer for the reason that if they are allowed to remain in the solution they tend deleteriously to affect the baking and keeping qualities of the yeast, causing rapid discoloration thereof and rendering it generally objectionable for bakers' use, while some are of such a nature as to render the molasses very difficult of filtration. Moreover, cane molasses is largely deficient in substances which are considered to be highly desirable in the commercial manufacture of yeast as for example, yeast-assimilable nitrogen, potassium, and phosphorus.

By the use of the present invention not only are the substances detrimental to the yeast-nutrient solution and which have heretofore been found to be quite difficult of removal, effectively removed from the solution, but the solution is simultaneously enriched to a considerable degree in the above mentioned substances which are so desirable from the standpoint of yeast manufacture.

According to the present invention, the clarification of the molasses is accomplished by diluting the same and rendering it distinctly alkaline by the addition thereto of suitable alkali materials or a mixture of such materials, containing one or more components which are readily assimilable by yeast, and in which the raw material is deficient, such as, for example, the hydroxides or carbonates of ammonium or potassium or mixtures thereof, and adding thereto a suitable substance capable of forming therein a phosphate precipitate hereinafter referred to as a "phosphate precipitant", as for example, phosphoric acid or the phosphates of ammonium or potassium, whereupon the molasses is heated moderately to complete the formation of the precipitate and is filtered in any suitable manner with or without the aid of the well known aids to filtration, such as for example, kieselguhr, filtercel and the like.

The following is a specific example of the method of carrying out the process:

The molasses is slightly diluted with water to the amount of about 2 parts of water to 1 part of molasses by volume, although a greater dilution may be used if desired. To the diluted molasses sufficient aquaammonia is added to render the same distinctly alka-

line, as for example, to a degree such that 2.0 cc. normal sulphuric acid solution will be required to neutralize 100 cc. of the alkaline molasses. The quantity of alkali substances required for this purpose may vary somewhat for different batches of cane molasses in view of the fact that the latter as usually obtained on the market possesses varying degrees of acidity.

After the diluted molasses has been rendered alkaline as described, there is added to it a suitable quantity of mono-ammonium-dihydrogen phosphate ($NH_4H_2PO_4$) as for example, about 1% of the original weight of the molasses under treatment before dilution. The phosphate is preferably added to the diluted molasses in the form of an aqueous solution of any convenient strength, and after its addition the mass is mixed thoroughly and heated moderately to a temperature sufficient to complete the precipitation and clarification, as for example, to from between 80° C., and the boiling point. This heating not only tends to promote the desired reaction between the added agents and certain constituents of the molasses, but also tends to sterilize the nutrient medium and possibly cause some modification of the constituents thereof so as to render them more readily assimilable when the solution is subsequently used in yeast manufacture.

After the heating has been continued for a sufficient time, as for example, from 1 to 2 hours the solution is allowed to stand until the larger part of the precipitate has settled whereupon the supernatent liquid is drawn off and filtered by any suitable means, as for example, through an ordinary filter press or a Sweetland filter preferably with the use of one of the usual filtering aids such as filtercel, kieselguhr, etc., used for example to the amount of about .5%.

Obviously the above procedure is merely illustrative of the way in which the present invention may be carried out and the materials used can be varied in the manner above set forth, for example, other alkaline compounds of ammonium or potassium or a mixture of such compounds may be used to render the raw materials alkaline and other suitable phosphorus containing materials such as phosphoric acid or potassium phosphate may be used to aid in the formation of the precipitate, the selection of the materials to be used being governed in accordance with the deficiencies of the raw material in the yeast-assimilable constituents afore-mentioned.

The present invention by one and the same treatment accomplishes not only elimination from commercial cane molasses of substances which tend to cause discoloration of yeast produced therefrom, but also may serve to introduce into the purified sacchariferous product at least a considerable proportion of the total quantity of yeast-assimilable nitrogen (and yeast-assimilable potassium also, if desired) necessary to constitute a well proportioned yeast nutrient with the sugar present. This enrichment of the purified molasses with yeast-assimilable nitrogen will take place as described notwithstanding the fact that some of the ammoniacal nitrogen which may be added in the form of ammonia or ammonium phosphate may enter into the composition of the precipitate which is separated from the purified molasses solution.

The yeast nutrient solution thus obtained may then be slightly acidified, for example, with sulphuric acid and may be either wholly or partially diluted to any suitable concentration and employed as a yeast nutrient solution, in any of the well known methods of manufacture utilizing aeration, for the commercial manufacture of bakers' yeast of good quality.

Moreover, if desired, the yeast-assimilable nitrogen content of the cane molasses purified and modified in accordance with my process taken either alone or with added malt sprouts, may be further augmented by causing to be commingled therewith further amounts of suitable substances containing yeast-assimilable nitrogen such as, for example, ammonium salts (as ammonium lactate, sulfate, phosphate, or carbonate), or compounds of an organic nature such as urea and the like. Obviously the commingling of the ammonium lactate may be accomplished if desired, by a controlled propagation of lactate acid bacteria in the solution with accompanying neutralization of the acid formed, by alkaline ammonium compounds, in any case the object being to form a yeast food well balanced with respect to its content of carbohydrate material and substances containing yeast-assimilable nitrogen and phosphorus.

When a bakers' yeast of a type or species such as is ordinarily employed in commercial yeast production is propagated in an aerated nutrient solution prepared in accordance with my present invention a yeast is obtainable on large scale commercial production which is of fully satisfactory keeping quality and baking strength and is fully satisfactory for marketing as a compressed bakers' yeast.

Since certain changes may be made in carrying out the above method without departing from the scope of the invention, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

It is also to be understood that the following claims are intended to cover all of the generic and specific features of the invention herein described, and all statements of the scope of the invention which, as a

matter of language, might be said to fall therebetween.

This application is in part a continuation of my copending application, Serial No. 597,145, filed October 26, 1922.

Having described my invention, what I claim as now, and desire to secure by Letters Patent, is:

1. A method of treating molasses adapted for use in the fermentation industries, which consists in rendering the molasses distinctly alkaline with an alkali compound containing yeast-assimilable components in which the molasses is deficient, adding thereto a substance adapted to form a phosphate precipitate, heating the mixture and thereafter separating the solution from the substances undissolved therein.

2. A method of treating molasses for use in the commercial manufacture of yeast by an aeration process which consists in rendering the molasses distinctly alkaline with an alkali compound containing yeast-assimilable components, adding thereto a phosphate precipitant, heating the mixture moderately and thereafter separating the solution from the substances undissolved therein, the alkali substance and the phosphorus containing substance being so selected and proportioned as materially to supply deficiencies of yeast-assimilable substances in the molasses.

3. A method of treating cane molasses for use in the commercial manufacture of yeast by an aeration process which comprises rendering the molasses distinctly alkaline with an alkali compound containing yeast-assimilable components in which the molasses is deficient, adding thereto a water-soluble phosphorus containing substance adapted to form a precipitate therein, heating the mixture moderately and thereafter separating the solution from the substances undissolved therein, the alkali and precipitating substances being selected from compounds of ammonium, potassium and phosphorus in such proportions as materially to supply the deficiencies of yeast-assimilable substances in the molasses.

4. A method of treating cane molasses for use in the manufacture of yeast by an aeration process, which comprises treating cane molasses with sufficient amounts of alkaline ammonium and potassium compounds to render it distinctly alkaline, adding thereto a water soluble phosphate containing essentially yeast-assimilable constituents, heating the mixture to approximately 80° C., and thereafter separating the solution from the substances undissolved therein.

5. A method of treating cane molasses for use in the manufacture of yeast by an aeration process, which comprises treating cane molasses with sufficient amounts of alkaline ammonium compounds to render it distinctly alkaline, adding thereto a water-soluble phosphate containing essentially yeast-assimilable constituents, heating the mixture to approximately 80° C., thereafter separating the solution from the substances undissolved therein.

6. A method of treating cane molasses for use in the manufacture of yeast by an aeration process, which comprises treating the molasses with sufficient ammonia to render it alkaline, adding an ammonium phosphate and thereafter separating the solution from substances undissolved therein.

7. A method of treating cane molasses for use in the manufacture of yeast by an aeration process, which comprises treating the molasses with sufficient ammonia to render it alkaline, adding an ammonium phosphate, heating the mixture to approximately 80° C., and thereafter separating the solution from substances undissolved therein.

8. A method of treating cane molasses for use in the manufacture of yeast by an aeration process, which comprises treating the molasses with sufficient ammonia to render it alkaline, adding a quantity of ammonium dihydrogen phosphate equivalent to about 1 per cent of the weight of the molasses originally taken for treatment and thereafter separating the solution from substances undissolved therein.

9. A method of treating cane molasses for use in the manufacture of yeast by an aeration process, which comprises treating the molasses with sufficient ammonia to render it alkaline, adding a quantity of ammonium dihydrogen phosphate equivalent to about 1 per cent of the weight of the molasses originally taken for treatment, heating the solution to approximately 80° C., and thereafter separating the solution from substances undissolved therein.

10. A sacchariferous aqueous solution adapted to be used as a yeast nutrient medium in the production of bakers' yeast by an aeration process, said solution being free from substances causing discoloration of yeast and containing the water soluble constituents of the reaction products of cane molasses, ammonia present in sufficient amounts to render the solution distinctly alkaline and an ammonium phosphate.

11. A sacchariferous aqueous solution adapted to be used as a yeast nutrient medium in the production of bakers' yeast by an aeration process, said solution being free from substances causing discoloration of yeast and containing the water-soluble constituents of the reaction products of cane molasses, ammonia and an ammonium phosphate, together with an admixture of aqueous malt sprout extract.

12. A method of treating cane molasses for use in the manufacture of yeast by an aeration process, which comprises treating

cane molasses with re-agents containing yeast assimilable components and which are capable of both freeing the molasses from yeast-discoloring substances and of forming with the molasses residue a substantially balanced yeast ration and filtering the treated molasses.

13. A method of treating cane molasses for use in the manufacture of yeast by an aeration process, which comprises treating cane molasses with re-agents containing yeast assimilable nitrogen and phosphorus in such proportions as to free the molasses from yeast-discoloring substances and to form with the molasses residue a substantially balanced yeast ration, and filtering the treated molasses.

14. A method of treating cane molasses for use in the manufacture of yeast by an aeration process, which comprises rendering the molasses alkaline by the addition of an alkali compound containing yeast-assimilable nitrogen, precipitating from the molasses the yeast-discoloring substances by the addition of substances containing yeast-assimilable phosphorus, and thereafter separating the undissolved substances from the solution, such materials and re-agents being used in such proportions that the resultant medium comprises a substantially balanced yeast ration.

15. A method of treating cane molasses for use in the manufacture of yeast by an aeration process, which comprises rendering the molasses alkaline by the addition of an alkali compound containing yeast-assimilable nitrogen, precipitating from the molasses the yeast-discoloring substances by the addition of substances containing yeast-assimilable phosphorus, separating the undissolved substances from the solution and subsequently rendering the medium slightly acid by the addition of sulphuric acid.

In testimony whereof I affix my signature.

FRED RANSOHOFF.

DEFENDANT'S EXHIBIT D-9

Date Received
September 17, 1925.

Laboratory No. 2652.

C B 6 WASH NO. 17-4

Fermenter No. 16. Plant No. 1.

Sample	BALLING. before filtration	BALLING. after filtration	P_2O_5 in Beer. Grape 100cc	Alcohol in Beer (Proof spirit)	Dried Yeast Grams per Liter	Nitrogen in Dried Yeast	Calls.	Diff.
start								
1:15 P.M.	1.5	1.0	.080	0.0	4.67 grs.	8.61%	8398	
2:15 "	2.0	0.9	.075	0.0	5.30	8.42%	8538	140
3:15 "	1.8	1.0	.070	0.0	6.99	8.70%	9308	330
4:15 "	2.1	1.1	.050	0.0	9.18	8.75%	9788	480
5:15 "	2.4	1.3	.022	0.0	11.20	8.37%	9828	520
6:15 "	2.6	1.5	.006	0.0	13.92	8.42%	10468	640
7:15 "	2.9	1.6	.002	0.0	14.97	Spoiled	11208	740
8:15 "	3.2	1.8	.0015	0.0	16.60	8.91%	12000	792
9:15 "	3.3	1.7	.0010	0.0	16.90	9.09%	12000	
10:15 "	3.2	1.8	.0018	0.0	16.30	7.39%		

Balling tests by Mr. Browne.

P_2O_5 test by Mr. Roake.

Alcohol by Mr. Streng

Beer was filtered through alundum crucibles, by Mr. Browne.

Nitrogen tests by Mr. Browne.

CHARLES S. ROAKE,
Sept. 28, 1925.

CSR

DEFENDANT'S EXHIBIT D-9-A.

Date Received.
September 18, 1925.

Laboratory No. 2674.

C B & W A S H N O. 18-2.

Fermenter No. 15. Plant No. 1.

Sample	BALLING. before filtration	BALLING. after filtration	P_2O_5 in Beer. Grs. per 100 cc	Alcohol (Proof Spirit)	Dried Yeast Nitrogen Grams per liter.	in Dried Yeast	Cells.	Diff.
Start								
18. M.	1.52	1.00	.090	0.0%	5.348	8.52%	8530	
2 "	1.80	1.00	.085	0.2	6.087	9.15%	8660	130
3 "	1.78	1.02	.080	0.0	7.396	8.00%	8990	330
4 "	1.90	1.13	.070	0.0	7.476	9.66%	9390	400
5 "	2.15	1.35	.030	0.0	9.042	Spoiled	9990	500
6 "	2.35	1.35	.0055	0.8	12.347	8.39%	10510	620
7 "	2.66	1.40	.0018	0.0	13.735	Spoiled	11230	720
8 "	2.76	1.57	.0010	0.0	15.306	8.96%	12000	770
9 "	2.97	1.58	.0005	0.0	15.126	Spoiled	13000	

Alcohol tests by Mr. Strang.

Above filtered through alundum crucibles by Mr. Roake.

CHARLES T. ROAKE.
Sept. 26,1925.

C T R

DEFENDANT'S EXHIBIT D-9-B.

2674. September 21, 1925. Lab. Number 2637.

SPECIAL TEST OF "AB" Yeast

Fermenter #9 Plant #3.

Time	Gallons	Difference	Mg. before filtration	Mg. after filtration	Alcohol	Dried Yeast in 1 liter	Nitrogen in Dried Yeast
10 A.M.	10984		1.35	1.10	zero	5.00 grms.	8.40%
11 "	10320	65	1.35	1.10	"	6.20 "	9.51%
12 "	10924	165	1.75	1.30	"	8.25 "	8.54
1 P.V.	10734	300	1.95	1.25	"	9.45 "	8.40
2 "	10974	240	2.25	1.35	"	10.95 "	8.40
3 "	11194	310	2.70	1.70	"	11.30 "	8.25
4 "	11444	360	2.90	1.75	"	14.10 "	8.06
5 "	12054	410	3.20	1.90	"	16.30 "	7.94
6 "	12084		3.30	1.95	"	16.80 "	8.13
7 "	12054		3.30	1.95	"	16.82 "	8.04

Average Yo stere about 76%

Dr. Frei ~~ent.~~

Corrected copy
Charles C Roast.

DEFENDANT'S EXHIBIT D-9-C.

September 18, 1925. Lab. No. 2637.

SPECIAL TEST ON YEAST # 12-2
Form. #9 from Plant # 2, Peekskill.

Time	Gals.	Difference	Sltg. before filtration	Sltg. after filtration	Alcohol	Dried yeast in 1 liter	Nitrogen in Dried Yeast.	Time
10 A.M.	10296		1.35	1.10	zero	5.0 gms.	5.63%	10 A.M.
11 "	10350	65	1.35	1.10	"	6.20 "	5.72	11 "
12 "	10524	165	1.75	1.20	"	8.25 "	4.24	12 "
1 P.M.	10724	200	1.95	1.25	"	9.45 "	5.25	1 P.M.
2 "	10974	250	2.25	1.35	"	10.95 "	5.25	2 "
3 "	11284	310	2.70	1.70	"	11.20 "	5.36	3 "
4 "	11644	360	2.90	1.75	"	14.10 "	6.04	4 "
5 "	12054	410	3.20	1.90	"	16.30 "	4.88	5 "
6 "	12054		3.30	1.95	"	16.90 "	5.88	
7 "	12054		3.30	1.95	"	14.82 "	5.04	

Moisture about 76%

Dr. Fred Peet.

Chas E Roake
Sept 15, 1925

DEFENDANT'S EXHIBIT D-9-D.

September 3, 1925. LAB. NO. 3612.

CB5

SPECIAL TEST ON STOCK MASH #6-4.
Form. #32 from Plant #2, Peekskill.

Time	Sale.	Difference	Mfg. before filtration	Mfg. after filtration	Alcohol	Dried Yeast in 1 liter		Nitrogen in Dried Yeast
10 A.M.	10283		1.70	1.30	sore	7.7 gms.		8.82
11 "	10473	-160	1.75	1.30	"	50% 4.8	= 1.3	8.58
12 "	10663	210	1.75	1.30	.2% Peroxide	36% 5.4	= 1.2	9.23
1 P.M.	10963	300	1.75	1.30	.2% "	6.6	= 1.2	9.30
2 "	11253	310	1.90	1.35	.4% "	34% 8.3	= 1.7	9.25
3 "	11613	360	2.00	1.40	.4% "	25% 10.4	= 2.1	9.25
4 "	12013	420	2.45	1.60	.2% "	17% 12.2	= 1.8	9.38
5 "	12613		2.45	1.60	sore	6.5% 83.8	= .80	9.23

Average Moisture 76%

15.9% N.

Dr. Fred Peet.

Charles Reed
Sept 8, 1925

14
4.5%

16

DEFENDANT'S EXHIBIT D-9-E.

Lab. No. 2305.

August 28, 1925.

SPECIAL TEST ON WASH # 28-S
Form. # 18. Plant # 3.

Time	Galls.	Diff.	Boiling before fermentation	Boiling after fermentation	Alcohol Dried Yeast in 1 liter	Dried Yeast
9 A.M.	10363		1.60	1.10	sore 4.10 grs.	9.27%
10 "	10441.5	78.5	1.60	1.15	= 30/8.30 = 1.20	9.07
11 "	10502	139.5	1.80	1.20	= 37/9.10 = 1.80	9.27
12 "	10795	293.0	2.05	1.30	= 284/9.10 = 2.00	8.96
1 P.M.	11086	291.0	2.35	1.30	= 104/10.00 = .90	9.36
2 "	11315	229.0	2.65	1.65	= 13/11.20 = 1.30	9.07
3 "	11677	362.0	2.95	1.85	= 9.7/14.40 = 1.10	9.24
4 "	12058	378.0	3.15	1.95	= 14/16.45 = 2.05	8.96
5 "	12255	---	3.30	1.95	= 3/16.95 = .506	9.16

Dr. Fred Peat.

Moisture Average about 75.5%

.34 Charles Roads aug 28, 1925

53) 180 (3
159
210
211

11) 1100 (100
1017
83
10
144) 2050 (14
1440
610
576

DEFENDANT'S EXHIBIT D-9-F.

Date Received.
September 15, 1925.

Laboratory No. 2672.

C B 6 W A S H NO. 15-3.

Fermenter No. 16. Plant No. 1.

Sample	BALLING. before filtration	BALLING. after filtration	P_2O_5 in Beer Gms. per 100 c.c.	Alcohol in Beer (Proof Spirit)	Dried Yeast Gms. per Liter	Nitrogen in Dried Yeast	Gals.	Diff.
at start	1.55	1.10	.070	0.0%	3.2898	9.26%	8580	
1st hr.	1.56	1.05	.050	0.2%	4.6964	8.49%	8880	130
2nd "	1.65	1.16	.045	0.2	4.5081	9.77	8990	330
3rd "	1.80	1.30	.053	0.2	6.7656	9.68	9390	400
4th "	2.65	1.35	.028	0.0	8.7460	9.32	9890	500
5th "	2.85	1.40	.023	0.0	10.7787	9.26	10510	620
6th "	2.56	1.54	.004	0.0	11.5822	9.20	11230	720
7th "	2.65	1.61	less than .002	0.0	10.2354 (c)	9.27	12000	770
8th "	2.75	1.55	less than .002	0.0	13.1167	spoiled	12000	
	Above tests by Mr. Tomkin		Tested by Mr. Strang		Tested by Mr. Reake.		Tested by Mr. Reake.	

In 7th hour high balling after filtering and apparently low yield of yeast were probably caused by an imperfect filtration as the buchner funnels are not very efficient. In the latter experiments similar crucibles were used to secure a better filtration.

CHARLES E. REAKE.
Sept. 26, 1925.

CLR

DEFENDANT'S EXHIBIT D-9-G.

September 14, 1925. Laboratory No. 2671

WASP CB 14-3.

REMEDIES NO. 16. PLANT NO. 1.

Sample	BALLING. before filtration	after filtration	Alcohol (Proof Spirit)	Dried Yeast Gr. per liter	Nitrogen in Dried Yeast	Galls.	Diff.
at start	1.50	1.05	0.0%	2.8900 gr.	7.89% N	8530	
1st hr.	1.55	1.02	0.2%	4.4812 gr.	8.96% N	8660	130
2nd "	1.55	1.05	0.2	5.4593	9.72%	8990	330
3rd "	1.65	1.30	0.0	6.3013	9.70%	9390	400
4th "	2.0	1.37	0.0	7.1557	9.38%	9890	500
5th "	2.30	13.6	0.0	9.6087	9.25%	10510	620
6th "	2.46	1.75	0.0	10.1807	9.29%	11230	720
7th "	2.70	1.75	0.0	12.3675	8.69%	12000	770
8th "	2.75	1.55	0.0	14.1763	8.52%	12000	

Alcohol Tests
by
Mr. Strang.

Charles T. Rents.

Sept. 26, 1925.

CER

DEFENDANT'S EXHIBIT D-9-H.

24

Date Received
Sept. 17, 1925.

Laboratory No. 3673.

3671

C B & WASH NO. 17-3

Fermenter No.15. Plant No. 1.

Sample	BALLING. before filtration	BALLING. after filtration	P.O.in Beer gr per 100 cc	Alcohol in Beer (Proof Spirit)	Dried Yeast Grams per Liter	Nitrogen in Dried Yeast	Gallo.	Difference.
Start	1.50	1.05	.090	0.0	3.7680	9.35%	8398	
1st hr.	1.65	1.00	.080	0.0	4.9994	10.07%	8838	140
2nd "	1.65	1.05	.075	0.0	5.3200	10.21%	8888	350
3rd "	1.77	1.25	.072	0.0	6.1526	10.14%	9308	420
4th "	2.10	1.30	.060	0.0	7.7500	9.96%	9828	520
5th "	2.26	1.35	.025	0.0	9.3030	9.71%	10468	640
6th "	2.97	1.36	.005	0.0	12.0800	9.51%	11208	740
7th "	2.87	1.65	.003	0.0	13.5200	Spoiled	12000	793
8th "	3.05	1.70	.0038	0.0	13.8800	8.73%	13000	
9th "	3.05	1.76	.0015	0.0	14.7580	8.30%		

Alcohol tests
by
Mr. Strang.

CHARLES V. NOAKY.

Sept. 26, 1925.

C & R

DEFENDANT'S EXHIBIT D-10.

Chart A

pH scale and Hydrogen ion concentration

pH		H ions (in units of grams x 10^{-7} per liter)
3.0		10,000
3.1 →		← 7943
3.6		3,163
4.0		1,000
Dr Lamb. .27 { 4.23 →		← 590
4.5		316
.1 { 4.6 →		← 251
Dr Fuller		
5.0		100
5.5		32
6.0		10
7.0		1

Dept of E

DEFENDANT'S EXHIBIT D-11.

Record of Prior Trials Including Pleadings and Exhibits.

(Physical Exhibit.)

STIPULATION AS TO RECORD.

It Is Hereby Stipulated and Agreed, by and between counsel for the respective parties hereto, that the foregoing transcript in two volumes is a true transcript of the record of the United States District Court for the District of New Jersey, in the above-entitled case, as agreed by the parties.

.....

Of Counsel for Plaintiff-Appellant.

Dated:

.....

Of Counsel for Defendant-Appellant.

Dated:

CLERK'S CERTIFICATE.

I, GEORGE T. CRANMER, Clerk of the District Court of the United States of America for the District of New Jersey, do hereby certify that the foregoing is a correct transcript of the record (in two volumes) of the said District Court in the above-entitled matter as agreed on by the parties.

IN TESTIMONY WHEREOF, I have caused the seal of said Court to be hereunto affixed, at the City of Trenton, in the District of
(Seal) New Jersey, this day of , in the year of our Lord one thousand nine hundred and thirty-eight, and of the Independence of the said United States the one hundred and sixty-second.

GEORGE T. CRANMER,
Clerk.

CLERK'S CERTIFICATE.

UNITED STATES OF AMERICA,
EASTERN DISTRICT OF PENNSYLVANIA, } *Sct.:*
THIRD JUDICIAL CIRCUIT.

I, Wm. P. Rowland, Clerk of the United States Circuit Court of Appeals for the Third Circuit, DO HEREBY CERTIFY the foregoing to be a true and faithful copy of the original Transcript of Record, Volume II (Exhibit Book) in the cases of Standard Brands, Inc., Plaintiff-Appellant, v. National Grain Yeast Corp., Defendant-Appellee, No. 6651; National Grain Yeast Corp., Defendant-Appellant, v. Standard Brands, Inc., Plaintiff-Appellee, No. 6670, on file, and now remaining among the records of the said Court, in my office.

IN TESTIMONY WHEREOF, I have hereunto subscribed my name and affixed the seal of the said Court, at Philadelphia, this twentieth day
(Seal) of February, in the year of our Lord one thousand nine hundred and thirty-nine and of the Independence of the United States the one hundred and sixty-third.

WM. P. ROWLAND,
Clerk of the U. S. Circuit Court of Appeals, Third Circuit.

SUPREME COURT OF THE UNITED STATES

ORDER ALLOWING CERTIORARI—Filed March 27, 1939

The petition herein for a writ of certiorari to the United States Circuit Court of Appeals for the Third Circuit is granted, limited to the question of the validity of Hayduck Patents Nos. 1,449,103; 1,449,105; and 1,449,106.

And it is further ordered that the duly certified copy of the transcript of the procedings below which accompanied the petition shall be treated as though filed in response to such writ.